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## DEMONSTRATION OF STEAM INJECTION AS AN ENHANCED SOURCE REMOVAL TECHNOLOGY FOR AQUIFER RESTORATION

## **Technical Report**

## Prepared for:

United States Air Force Research Laboratory Materials and Manufacturing Directorate Airbase and Environmental Technology Division Armstrong Laboratory, USAFRL/MLQE 139 Barnes Drive, Suite 2 Tyndall Air Force Base, Florida 32403-5323

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Utah. The purposes of the research were to increase understanding of the technology and evaluate its effectiveness for cleanup of nonaqueous phase liquids (NAPLs). The results will be included in an evaluation of various technologies for potential inclusion in the OU-1 Record of Decision. Applied Research Associates, Inc. and Praxis Environmental Technologies, Inc. teamed to conduct the experiments in a 3-by-5 meter cell enclosed by steel sheet-pile walls extending into a low permeability clay layer located about 9.1 meters (30 feet) below ground surface. Constituents of the NAPL were primarily weathered fuels and oils with chlorinated solvents. Results of the steam injection test reveal very effective distillation of contaminants from the residual NAPL. Compounds of concern showed reductions in soil concentrations greater than 95% in steam-swept layers. The final groundwater concentrations in the test cell were close to cleanup levels with only two target compounds exceeding the drinking water standard. As predicted by theory, the heavy NAPL was not effectively mobilized by the steam because of its high viscosity and low residual saturation; yet the low residual saturation allowed good contact between the steam and NAPL for distillation. Estimates of the masses removed based on extracted concentrations were consistent with mass changes in the cell based on soil concentrations. An estimated total of 55 liters (14.5 gallons) of NAPL were removed from the cell. This study also employed an innovative technique known as a partitioning inter-well tracer test (PITT) to estimate the mass and spatial distribution of residual NAPL before and after the steam injection. The PITT consists of the simultaneous injection of several partitioning and non-partitioning tracers into injection wells and the measurement of tracer concentrations at various downgradient monitoring points. Data analysis of the pre-steam injection PITT indicate an average NAPL saturation of about 5% and an estimated 394 liters (104 gallons) of NAPL in the saturated zone of the test cell. Results of the post-steam injection PITT are pending at this time.

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## **PREFACE**

This report was prepared by Applied Research Associates, Inc., New England Division, RR#1, Box 120-A, Waterman Road, South Royalton, VT 05068, and PRAXIS Environmental Technologies, Inc., 1440 Rollins Road, Burlingame, CA 94010, under Contract Number F08635-93-C-0020, Subtask 8.01.5 for the U.S. Air Force and the Armstrong Laboratory Environics Directorate (USAFRL/MLQE), 139 Barnes Drive, Suite 2, Tyndall Air Force Base, FL 32403-5323.

This technical report describes the background, methodology, system design, and results of a steam injection/vapor extraction treatability study conducted at the OU-1 site, Hill Air Force Base, Ogden, Utah.

The authors wish to acknowledge the technical and logistical support provided by Kevin Bourne and Jon Ginn of Hill AFB and Dr. Carl Enfield and Dr. Lynn Wood from the USEPA's Robert S. Kerr Laboratory (RSKRL).

The work was performed between April 1, 1995 and March 31, 1997. The AL/EQM project officers were Captain Jeffrey Stinson and Major Paul Devane.

#### **EXECUTIVE SUMMARY**

## A. OBJECTIVE

Steam injection, combined with soil vapor extraction, was demonstrated in situ at Operable Unit One (OU-1), Hill Air Force Base (Hill AFB), Utah. The purpose of this research was to evaluate steam injection technology for the removal of non-aqueous-phase liquid (NAPL) contamination from the subsurface. This experiment was part of a cooperative research effort funded by the United States Environmental Protection Agency (USEPA) and the Strategic Environmental Research and Development Program (SERDP). The results of this research were used to evaluate eight innovative remediation technologies for the removal of NAPL and to evaluate these technologies for their potential inclusion in the Record of Decision (ROD) for the OU-1 site. Applied Research Associates, Inc. (ARA) and Praxis Environmental Technologies, Inc. demonstrated the utility of steam injection combined with soil vapor extraction as a remediation technique. In addition, the experiment included the use of a partitioning tracer test, which was employed to estimate the quantity and distribution of the NAPL prior to and immediately following the steam treatment.

#### B. BACKGROUND

Subsurface contamination by NAPLs, such as hydrocarbon fuels and halogenated organic solvents, is a serious environmental problem facing the Department of Defense and industry in general. Once the NAPLs migrate into the subsurface environment, significant quantities of the liquid become trapped in the soil by capillary forces, providing a continuous source of groundwater contamination. Complete removal of these contaminants by conventional technologies is difficult, time-consuming and expensive. Historically, technologies such as pump-and-treat and soil vapor extraction have been used with moderate success, but in general, they are inefficient and costly technologies to achieve the desired clean-up goals. Recent studies have demonstrated the utility of using steam injection and subsequent vacuum extraction of steam to remove NAPL contamination from in situ soils.<sup>1</sup>

The use of steam injection to remediate NAPLs and saturated zone contamination is an innovative application of a recently developed remedial technology. Many of the technology principles have been tested extensively in analogous field applications (e.g., enhanced oil recovery). However, steam injection has been applied only recently to the remediation of shallow subsurface contamination where required recovery rates are much higher. Most of this recent experience applies to soil above the water table and these efforts were not instrumented adequately for understanding the process. Hence, this project will be a carefully monitored and documented effort to advance the understanding and the state of design of steam injection.

## C. SCOPE

This document presents and analyzes the findings of the steam injection/vapor extraction treatability experiment conducted at the OU-1 site located at Hill AFB, Ogden, Utah. Section I is

<sup>&</sup>lt;sup>1</sup> Stewart and Udell, 1987, and Olsen et al., 1991.

an introduction to the technology and includes a brief literature review, description of the site, and the project objectives. Section II presents an overview of the methodology followed during the course of the study and includes descriptions of the test cell construction, leak testing, and pre- and posttreatment cell characterization. It also includes a conceptualized narrative of the steam injection/vapor extraction system. Section III includes detailed descriptions of the processes and procedure followed during the test execution for all phases of the experiment. This section includes discussions about both the fluorescein dye and the partitioning interwell tracer tests, the steam injection process equipment and finally, the implementation of the steam injection experimental phase's. The results of the experimentation are presented in Section IV and finally the conclusions and recommendations are presented in sections V and VI, respectively.

#### D. METHODOLOGY

To achieve the goals of this experimental research, the project was divided into two phases. Phase 1 consisted of the cooperative development of a workplan by all of the individual research groups, the EPA, and Hill Air Force Base. It also involved some preliminary bench scale testing by the researchers to aid in the experimental design. Preliminary field investigations were also conducted during Phase I to collect site characterization data to assist with cell placement. Finally, Phase I included the installation of the eight individual experimental cells.

Phase II consisted of several subtasks. First, an extensive characterization effort was conducted for each of the individual cells. This characterization included the collection and logging of soil samples, and subsequent chemical analysis of these samples. These data provided an estimate of the pretreatment NAPL saturation and distribution, and served as a baseline to compare to the posttreatment results. Coinciding with the cell characterization activities, was the installation of the multilevel sampling points, steam injection/extraction wells, and piezometers.

Once cell construction activities were completed, a partitioning interwell tracer test (PITT) was conducted as an additional method to estimate the saturation and distribution of the NAPL within the cell prior to steam treatment. Groundwater samples were collected before and after the PITT to determine the static and dynamic equilibrium concentrations of the target analytes in the groundwater.

Following the characterization efforts, the steam treatment activities commenced, consisting of five individual stages: (1) dewatering the cell, (2) pre-steam-injection soil vapor extraction, (3) steam injection, (4) post-steam-injection soil vapor extraction, and (5) reflooding the cell and cooling.

Once the cell cooled to the pretreatment in situ temperature, a posttreatment PITT was conducted to estimate the residual saturation of the NAPL within the cell. These data were used as a secondary method to estimate the efficiency of NAPL removal from the cell. As with the pretreatment PITT, groundwater samples were collected under dynamic (prior to the PITT) and static (after the PITT) conditions.

Finally, the posttreatment characterization was conducted to collect soil samples for chemical analysis and to further define the lithology of the cell. All of the data associated with this experiment is presented in this report with the exception of postdemonstration PITT analysis. This analysis was conducted by others and was not complete as of this date.

## E. TEST DESCRIPTION

## 1. Partitioning Interwell Tracer Test (PITT)

The PITT test consists of the simultaneous injection of a slug of dilute concentrations of both partitioning and non-partitioning tracers into the test cell under constant flow conditions. The tracer solution is subsequently sampled through the MLS sampling grid and extraction wells. The non-partitioning tracers flow through the cell unimpeded by the NAPL, behaving much like a particle of water. The partitioning tracers interact with the NAPL, moving in and out of the NAPL solution at a rate proportional to the NAPL/tracer partitioning coefficient. The net result of this interaction is that the partitioning tracers are retarded with respect to the non-partitioning tracers. By plotting concentration breakthrough curves for each tracer and comparing the first moments (mean residence time) of each, an estimate of the magnitude and distribution of NAPL can be determined.

## 2. Steam Injection / Vapor Extraction Treatment

The steam injection/vapor extraction test consisted of injecting steam into vertical injection wells placed within the region of contamination and subsequent removal from the extraction wells placed within and around this region. First, relatively high pressure gradients develop in the steam zone due to the high vapor velocities. These pressure gradients force the effective displacement of original water and contaminant in place. Liquids that are "pushed" into the well are removed via pumps until steam breakthrough occurs. Application of a vacuum to the recovery wells during the injection of the steam aids in directing flow of steam toward the extraction wells through the vadose zone, and contaminant recovery is identical to that of soil vapor extraction technology until steam breakthrough. After breakthrough, the steam vapor behaves like air during soil venting, only now the soil is at an elevated temperature. The vapor pressures of typical organic compounds increase by factors from 25 to 40 over those at ambient soil temperatures. This greatly accelerates evaporation rates and reduces remediation duration.

## F. RESULTS

Based on visual descriptions of the soil corings collected during the pre- and posttreatment characterization activities, a detailed three-dimensional model of the soil stratigraphy was developed. The model shows clearly that there are four distinct stratigraphic sections within the cell consisting of three interbedded soil types; (1) poorly graded sands, (2) well-graded gravelly sand mix, and (3) clay. Chemical analyses of the soil core samples show elevated concentrations of all of the target analytes within the cell with the highest levels located at about (18-20 feet) below grade. This coincided with the lithologic section with the highest hydraulic conductivity.

Data analysis of the pre-steam injection PITT using the method of moments technique indicates an average NAPL saturation of about 5 percent and an estimated 469 liters (124 gallons) of NAPL in the saturated zone of the test cell. The results from the method of inverse modeling support the results obtained using the method of first temporal moment analysis. Both methods indicate that the average NAPL saturation in the test cell is approximately 5 percent. However, due to a slightly different cell geometry assumed for the method of inverse modeling, the volume of NAPL estimated was 394 liters (104 gallons).

Based on the results of the pre- and posttreatment groundwater chemical analyses, the average percent recoveries of the target analytes ranged from 88 percent removal (1,1,1-trichloroethane) to 28 percent removal (TCE). Concentrations of several of the less volatile compounds increased significantly (88 percent increase for 1,2-dichlorobenzene). This result occurred because of changes in the NAPL makeup. As the more volatile compounds are stripped out, the less volatile compounds, such as 1,2-DCB are left at a higher mole fraction. The higher mole fraction then yields a higher equilibrium groundwater concentration despite significant removal of the compound. Yet, only two of 15 target compounds (TCE and 1,2-DCB) were above drinking water standards at the end of the test.

The mass of target compounds removed during the pre-steam SVE, steam injection and poststeam SVE tests was estimated from the measured extraction rate and the measured concentrations. These results are summarized below:

Phase	Extracted Volume of Air (m³)	Extraction Period (hours)	Average Total Target Compound Concentration (mg/m³)	Total Mass Removed (kilograms)
Pre-Steam SVE	2030	47	445	0.9
Steam Injection	3194	100	1900	6.0
Post-Steam SVE	24,260	356	106	2.6

Analysis of the posttreatment PITT results is being conducted by others and is currently not available. This analysis, along with a discussion comparing the results to the chemical analytical results of the soil cores, will be included as an addendum to this document upon receipt.

#### G. CONCLUSIONS

The total NAPL volume estimated from the method of first moment analysis was determined to be approximately 469 liters (124 gallons). This value was obtained by tracer data extrapolation up to 16 days. It represents the NAPL volume in the saturated zone of the entire test cell, which corresponds to an estimated tracer-swept volume of 9.3 m<sup>3</sup>. Because of the irregularly shaped boundary of the test cell, the simulation grid for the inverse modeling technique only represents the rectangular portion of the test cell between the rows of the injection and extraction wells. The estimated volume of NAPL within this region is 394 liters (104

gallons). Assuming a porosity of 0.28, this contains a pore volume of 8.19 m<sup>3</sup>. For both scenarios, the ratio of volume of NAPL to volume of pore space is approximately 50 liters/m<sup>3</sup>. The NAPL is nonuniformly distributed in the test cell ranging from 0 to 10 percent in saturation. The average NAPL saturation is higher in the intermediate layers of the test cell.

The vapor concentrations of the more volatile compounds such as 1,1,1-trichloroethane (1,1,1-TCA) and heptane in the waste stream during the initial ambient soil vapor extraction (SVE) were initially high and exhibited the exponential decay characteristic of long-term SVE. For moderately volatile compounds such as toluene and nonane, the vapor concentrations appeared to decrease slightly during the tests. Concentrations of compounds with relatively low volatility, such as 1,2-dichlorobenzene (1,2-DCB) and undecane, were erratic and did not appear to decrease during the SVE testing.

Careful examination of the results from the SVE and steam injection portions of the remediation indicate disequilibrium of the NAPL (i.e., a nonuniform mixture) that would result from weathering of the NAPL over time or the presence of two distinct NAPL layers. The site usage history indicates two NAPL sources: one NAPL was the result of hydrocarbon usage for fire training, while the other NAPL resulted from chemical disposal pits that included the release of solvents. Approximately 34 kg (75 pounds) of NAPL were removed from the cell through the vapor stream during the course of the experiment. Assuming a unit weight of 0.75 g/cm<sup>3</sup> for the NAPL, this equates to about 45.5 liters (12 gallons). An additional 9.5 liters (2.5 gallons) were recovered in the NAPL/water separator.

The final soil and groundwater concentrations in the test cell were significantly reduced from the pre-test concentrations. Estimates of mass removed based on soil concentrations before and after steaming reveal over 90% removal for volatile compounds, 80 to 90% removal for moderately volatile compounds and 70 to 80% for semi-volatile compounds. In addition, soils swept directly by the steam exhibited excellent cleanup and the soils which were not swept showed reductions but not as profound. The steam swept soils were cleaned of the target compounds by over 94% including the semi-volatile compounds. A deeper steam sweep was not possible in this field test because the groundwater pump inlets could not be placed deeper than 6 m. It is expected the same high levels of removal would have been achieved in the lower soils if deeper screen and pump placement had been possible.

A bank of NAPL preceding steam breakthrough was considered possible; yet, only about 9.5 liters (2.5 gallons) of NAPL were recovered in the NAPL/water separator after steam breakthrough. This indicates the steam injection was not effective at driving the residual NAPL out of the cell. This occurred because the viscosity of the NAPL was too high and the saturation too low to allow the formation of a stable NAPL bank ahead of the steam condensation front. Theory predicting the maximum NAPL viscosity which allows a stable NAPL bank to mobilize was developed and suggested the maximum NAPL viscosity allowing stable displacement by steam injection at OU-1 is about 2.5 centipoise (cP). The NAPL at Operable Unit One has a viscosity significantly higher than 2.5 cP.

## H. RECOMMENDATIONS

Based upon the results obtained during the course of this project several recommendations can be made. The results from this study showed steam injection to be very effective in distilling contaminants from the mixed NAPL at OU-1, Hill AFB. Yet, the increase in vapor concentrations of the moderately volatile compounds was not as high as expected based upon the vapor pressures of these compounds at the elevated temperature. Further study is warranted to evaluate the reasons for this lack of increase. In particular, the role which liquid water may play in this process needs more investigation because further understanding could lead to substantial improvements in the technology implementation. Additionally, a substantial bank of NAPL was not pushed ahead of the steam condensation front in this demonstration. Theory was presented suggesting a relatively low limiting viscosity for such a push to occur. This theory requires additional laboratory and field studies for validation because of the potential impact this result could have on how the technology is applied to heavier hydrocarbons. Also, the evaluation of the technology for other contaminants and soil types should be pursued.

Any of these additional studies should also comment on the costs of using steam injection as a remediation technology. For coarse, gravely soils, such as those at Hill, the injection and extraction wells can be relatively far apart, whereas for fine grained soils, more wells per unit area may be required, driving the cost higher than experienced during this study. These costs need to be considered when performing a complete evaluation of steam injection remediation for a site. In addition, the feasibility of using pushed wells for injection and extraction of the steam should be studied. This well installation procedure has the potential to be faster, cheaper, and more informative without any loss in performance.

A second recommendation is to further enhance and develop the Partitioning Interwell Tracer Test (PITT). Although this test was very useful in determining the pre- and postcontaminant locations and saturation levels, performing the test was relatively expensive and very labor intensive. Additional methods to reduce the costs of performing these tests would greatly assist in increasing the utility of these tests. A new approach would still use partitioning tracers, but rather than collect samples over a 10-day period (over 2,000 samples were collected and analyzed for each PITT test during this demonstration), a monitoring system could be used to monitor the partitioning in-situ. This would require a sensor network to be installed and different tracers to be selected that matched with the sensing technology chosen for the network. One sensing technology that should be investigated is fluorescence techniques. If partitioning fluorescence sensors can be selected, then a network of simple fluorescence probes could be used to monitor the experiment. Since the sample collection rate would not be limited by actual sample collection time, more detailed results can be collected at low additional cost. A Cone Penetrometer fluorescence sensor version can be used to monitor the tracers in an open field condition, under a lower gradient. These approaches effectively reduce costs and allow more flexibility in the tracer flow field.

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## SECTION I INTRODUCTION

## A. OBJECTIVE

Subsurface contamination by non-aqueous-phase liquids (NAPLs), such as hydrocarbon fuels and halogenated organic solvents, is a serious environmental problem facing the Department of Defense and industry in general. Once the NAPLs migrate into the subsurface environment, significant quantities of the liquid become trapped in the soil by capillary forces providing a continuous source of groundwater contamination. Complete removal of these contaminants by conventional technologies is difficult, time-consuming and expensive. Historically, technologies such as pump-and-treat, and soil vapor extraction have been used with some success, but in general, they are inefficient and costly because the time required to operate these systems to achieve the desired clean-up goals is typically many years. Recent studies have demonstrated the utility of an alternative remediation technique involving the injection and subsequent vacuum extraction of steam to remove NAPL contamination from in situ soils (Stewart and Udell, 1987, and Olsen et al., 1991).

Steam injection, combined with soil vapor extraction, was demonstrated in situ at Operable Unit One (OU-1), Hill Air Force Base (Hill AFB), Utah. The purpose of this research was to evaluate steam injection technology for the removal of NAPL contamination from the subsurface. This experiment was part of a cooperative research effort funded by the United States Environmental Protection Agency (USEPA) and the Strategic Environmental Research and Development Program (SERDP). The results of this research were used to evaluate eight innovative remediation technologies for the removal of NAPL and to evaluate these technologies for their potential inclusion in the Record of Decision (ROD) for the OU-1 site. Applied Research Associates, Inc. (ARA) and subcontractor Praxis Environmental Technologies, Inc. (Praxis), under contract with the U.S. Air Force's Armstrong Laboratory (SETA contract No. F 8635 93 C0020 Subtask 8.01.5), demonstrated the utility of steam injection combined with soil vapor extraction as a remediation technique. In addition, the experiment included the use of

a partitioning interwell tracer test (PITT) that was employed to estimate the quantity and distribution of the NAPL prior to and immediately following the steam treatment.

#### B. BACKGROUND

## 1. Description of Technology

The use of steam injection to remediate non-aqueous-phase liquids (NAPLs) and saturated zone contamination is an innovative application of a recently developed remedial technology. Many of the technology principles have been tested extensively in analogous field applications (e.g., enhanced oil recovery). However, steam injection has been applied only recently to the remediation of shallow subsurface contamination where required recovery rates are much higher. Most of this recent experience applies to soil above the water table and these efforts were not instrumented adequately for understanding the process. Hence, this project will be a carefully monitored and documented effort to advance the understanding and the state of design of steam injection.

Thermal techniques to increase the recovery of volatile and semi-volatile liquids from porous media are not new. A large body of research on steam injection for enhanced oil recovery exists in the petroleum literature (Mandl and Volek, 1969, Volek and Pryor, 1972, and Konopnicki et al., 1979). This research concentrated on viscosity reduction and distillation. Recent research has investigated steam injection for light-oil recovery (Stewart and Udell, 1987, and Olsen et al., 1991). However, the use of thermal processes for the in situ recovery of contaminants has a short history. Exploratory field work on steam injection was performed in the Netherlands in the mid 1980s (Hilberts, 1985). Radio frequency heating (Dev, 1986) and in situ vitrification (Fitzpatrick et al., 1986) are other innovative thermal techniques under development. The combined steam injection and vacuum extraction process was laboratory-tested and field-demonstrated (Udell and Stewart, 1989) in 1989.

## 2. Site Description

Hill Air Force Base is located in northern Utah on a topographic plateau about 300 feet above the Weber River Valley (Figure 1). In July 1987, Hill AFB was placed on the National Priorities List (NPL). Eight operable units at Hill AFB have been identified; each of these

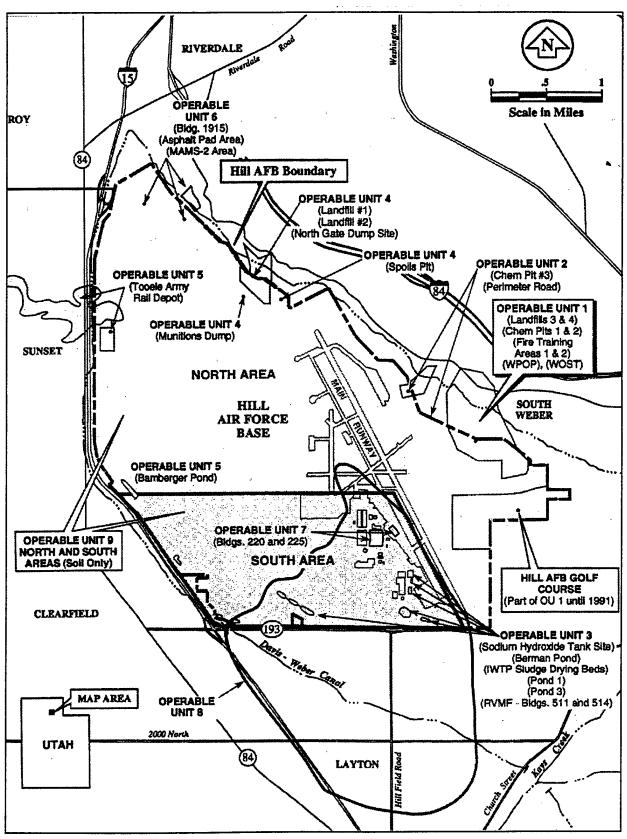


Figure 1. Site Location Map Depicting the Operable Units at Hill AFB.

operable units is associated with the past disposal of hazardous waste and consists of two or more waste sites. The description that follows is paraphrased from Montgomery Watson (1994).

Operable Unit 1 (OU-1) is located along the eastern boundary of Hill AFB, as shown in Figure 1, and includes the former Chemical Disposal Pits 1 and 2, Landfills 3 and 4, Fire Training Areas 1 and 2, the waste oil/phenol pit, and the waste oil storage tank. The primary area of concern for the steam injection demonstration is near the chemical disposal pits (Figure 2).

Many soil borings have been logged at the OU-1 site. Borehole depths ranged from a few feet to 120 feet below ground surface (bgs). Examination of the geologic logs from test wells and soil borings reveals that lithologic changes occur over very short distances both vertically and horizontally. The aquifer is composed of interbedded silts, sands, and gravels of the Provo Formation. Cross sections developed by Montgomery Watson indicate that an aquitard consisting of laminated clay with thin silt and sand interlaminae exists from depths of about 25 to 30 feet bgs. These deposits are characteristic of the Alpine formation. The top surface of the aquitard is very irregular and possibly represents an erosional surface on which the coarsergrained channel deposits of the Provo Formation were deposited.

Groundwater at OU-1 occurs approximately 20 to 25 feet below ground surface in the uppermost unconfined aquifer and flows to the northwest and west towards the Weber River Valley. This shallow aquifer is separated from deeper regional drinking water sources by about two hundred feet of principally low-permeability clays. During wet seasons, a number of seeps and springs occur off base on the face of the slope north of the site. Studies by Montgomery Watson indicate that the upper unconfined aquifer saturated thickness is only a few feet above the aquitard in the vicinity of the Chemical Disposal Pits.

An accumulation of light, non-aqueous-phase liquid (LNAPL) has been identified floating on the water table in the uppermost aquifer in the vicinity of Chemical Disposal Pits 1 and 2. The LNAPL accumulation is the result of fuel usage at Fire Training Area 1 and disposal practices at the Chemical Disposal Pits and Landfill 3. The LNAPL plume has migrated to the northwest under the Chemical Disposal Pits. The continued presence of the contamination is a threat to deeper regional drinking water aquifers.

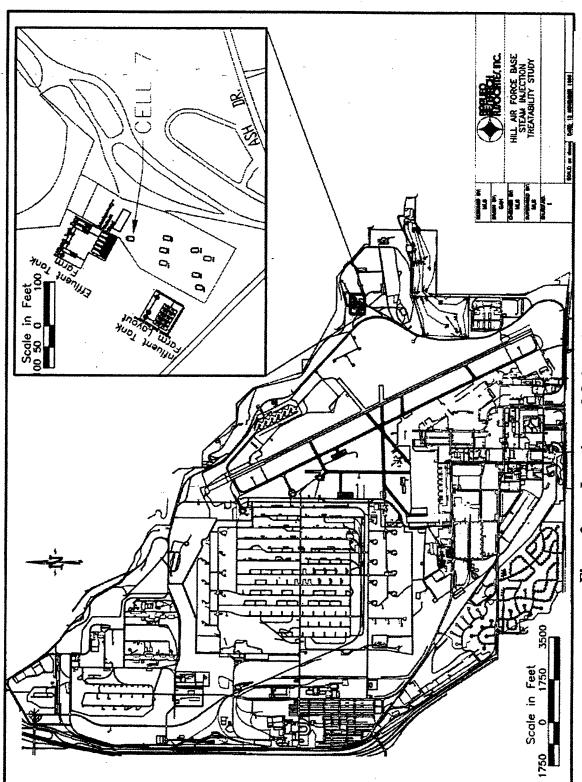


Figure 2. Location and Orientation of Study Area.

## C. SCOPE

Applied Research Associates, Inc. and Praxis conducted a controlled remediation demonstration using steam injection enhanced vapor extraction. This research effort is part of a collaborative study funded by the Strategic Environmental Research and Development Program (SERDP), the Advanced Applied Technology Demonstration Facility (AATDF) and the United States Environmental Protection Agency (EPA). This research was conducted at Operable Unit One (OU-1) located on Hill AFB, Ogden, UT.

The purpose of the SERDP Treatability Studies was to evaluate eight innovative remediation technologies for the removal of non-aqueous-phase liquid (NAPL) constituents from saturated and, in some cases, unsaturated soils, and to conduct treatability studies of these technologies for remediation of LNAPL contamination at Hill AFB Operable Unit One (OU-1). The evaluation was conducted by developing site-specific design information for each technology in the laboratory, then demonstrating the respective technologies in situ at a designated area at the OU-1 site. Each field demonstration was executed inside a test cell constructed to hydraulically isolate the cell volume and minimize migration into or out of the cell. In many cases, this was the first time some of these innovative technologies were demonstrated in the field.

Specifically, ARA/Praxis were tasked to develop and execute an experimental design employing vapor extraction enhanced by steam injection to remediate NAPL contamination in an isolated test cell.

An innovative technique known as a Partitioning Interwell Tracer Test (PITT) was used to establish the concentration levels and volumetric distribution of the NAPL within the test cell prior to and following the steam injection experiment. The test involved the injection of various alcoholic tracers, both partitioning and nonpartitioning into the test cell under constant hydraulic gradient. Subsequent sampling from a multilevel groundwater sampling grid and chemical analysis of the cell fluid provided individual breakthrough curves for each of the tracers. Mathematical analysis of the breakthrough-curve data using the method of moments and inverse modeling techniques provided an estimate of both the NAPL saturation and distribution within the test cell. Comparison of the saturation data prior to and following the steam injection

treatment yielded an estimate of the quantity of NAPL removed and some insight into the effectiveness of the remediation technique.

## D. OBJECTIVES

The overall objectives of the treatability studies were:

- To advance the development of these eight remedial technologies through research in the laboratory and demonstration in the field.
- To evaluate the relative performance of these eight technologies on a common basis for removing the NAPL constituents at Hill AFB.
- To obtain and document data of suitable quantity and quality to support evaluation of these innovative technologies in Feasibility Studies (FS) for inclusion in the Record of Decision (ROD) for the OU-1 site.
- To obtain design information necessary for scale up, cost estimating and implementation of the technologies at Hill AFB.

This report discusses the findings of the steam injection experiment only. A comprehensive report covering all eight remediation technologies will be compiled by the National Risk Management Research Lab (NRMRL), Subsurface Protection and Remediation Division in Ada, Oklahoma.

# SECTION II METHODOLOGY

## A. TEST CELL CONSTRUCTION

During the Spring of 1995, preliminary site characterization activities of the OU-1 site were conducted to determine suitable locations for the individual test cells. The criteria for locating the test cells were depth to clay, presence and extent of NAPL, depth to groundwater and logistical constraints. In addition, an effort was made to orient the cells so that the long axis of the cell ran parallel to the direction of the natural groundwater gradient at the OU-1 site. This allowed the experiments to be conducted, utilizing the naturally occurring in situ flowpaths.

The results of the preliminary investigation concluded that the underlying clay aquitard was at an acceptable depth in the area proximate to the Chemical Disposal Pit 2. Based on the data gathered during this preliminary investigation, the eight cells were sited and cell construction activities began in the Fall of 1995.

## 1. Test Cell Description

Experiments in test cells were conducted to isolate the test area from the surrounding aquifer to prevent mobilization or solubilization of contaminants that are currently immobile or slightly soluble under normal in situ conditions. The cells measured 3 by 5 meters (10 by 16 feet) (nominal) in plan view and extended approximately 9 meters (30 feet) into the earth, seating into the underlying Alpine Formation. The cells were constructed by driving interlocking steel sheet piles into the ground, forming a rectangular volume. The cell is sealed off beneath by an underlying clay aquitard, effectively eliminating downward migration out the bottom of the cell. Figure 3 illustrates the location and layout of the completed Cell 7 that was used during this experiment.

Standard, 9.5-mm (.37 inch) thick by 10.7-meter (35-foot) long, Z-shaped, cold-rolled, steel sheet piles with hook-and-grip interlocking joints were used to construct the test cells.

Angle iron was welded to the back of each sheet-pile joint to form a void that could be grouted to

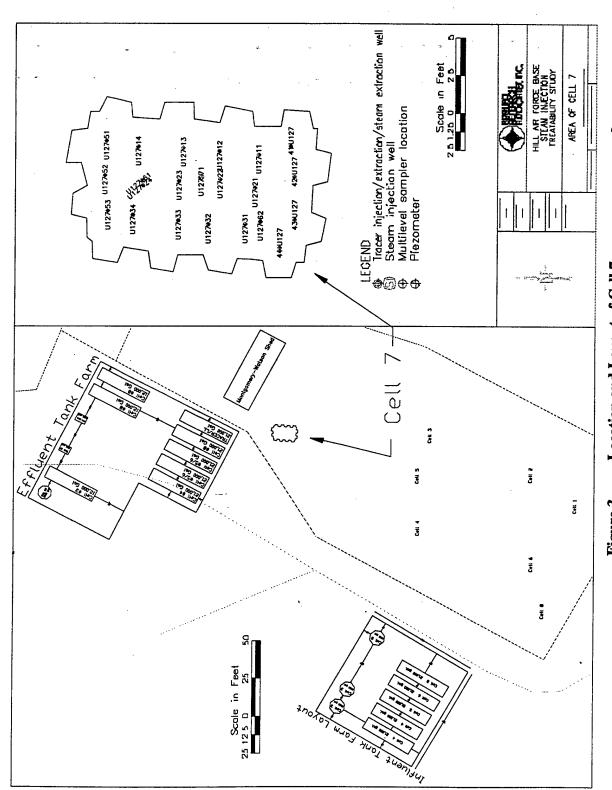


Figure 3. Location and Layout of Cell 7.

seal the joints. The piles were installed using a vibratory power drive hammer (APE Model 200 Vibro) attached to a crane. The individual piles for the steam injection test cell (Cell 7) were driven to the depths described in Table 1. Each joint was grouted and allowed to cure for approximately 30 days. After the cure period, all cells were tested as described below in § II.B.

TABLE 1. ESTIMATED SHEET PILE PENETRATION INTO THE SILTY CLAY LAYER FOR CELL 7.

	Steam Inject	ion			
Depth to clay: 27.0 ft 1					
Joint #	Cleared Joint Depth	Depth Into Clay Cleared			
	(ft)	(ft)			
1	31.5	4.5			
2	30	3			
3	32	5			
4	30	3			
5	20*	-7			
6	30	3			
7	30	3			
8	29	2			
9	30	3			
10	33	6			
11	30	3			
12	30	3			
13	35	8			
14	- 30	3			
15	30	3			
16	30	3			
17	30	3			
18	34	7			
19	30	3			
20	30.5	3.5			
21	30	3			
22	31	4			
23	30	3			
24	31	4			

<sup>&</sup>lt;sup>1</sup> According to Fugro's CPT logs, the clay does not appear to be a continuous unit.

<sup>\*</sup> Broke off bit - still in cavity.

#### 2. Well/MLS Construction

After the cell construction activities were completed and the grout allowed to set, preliminary cell characterization, and well/multilevel sampler (MLS) construction commenced. These activities began in November 1995, when ARA mobilized to the OU-1 site and, working closely with the EPA and the EPA's subcontractor, ManTech Environmental Services, Inc., installed the injection/extraction wells and the MLS sampling grid. The layout of the completed cell is shown in Figure 4 and shows:

- a bank of four injection wells (U1-2741 through U1-2744) on the south end of the cell,
- a bank of three extraction wells (U1-2751 through U1-2753) on the north end,
- one steam injection well (U1-2771) in the center of the cell,
- two piezometers (U1-2761 and U1-2762) on the respective north and south ends of the cell, and finally,
- 12 MLS sampling clusters (U1-2711 through U1-2734) forming a matrix evenly spaced between the injection and extraction wells.

#### a. MLS Construction

The MLS sampling clusters allowed for the collection of groundwater and/or soil gas samples from five discrete levels within the formation. There were 12 clusters, each fabricated from five different lengths of 3.2-mm (.12-inch) (OD), tempered stainless steel tubing with 100-microsintered stainless steel filters attached to the bottom end using stainless steel Swagelock® fittings. All tubing and filters were flushed with methylene chloride to remove any residual oils prior to construction of the MLSs. The five different lengths of tubing with attached filters were bunched together, staggering the filters every 0.6 meters (2 feet). The bunches were bound together using nylon snap ties at 0.3-meter (1-foot) intervals.

Once assembled, the MLS clusters were pushed into the cell using the US EPA's cone penetrometer technology (CPT) truck so that the deepest MLS filter was approximately 7 meters (23 feet) below ground surface. Each MLS bunch was sandpacked, then sealed at the surface with a bentonite seal. Installation of all of the MLSs created a three-dimensional

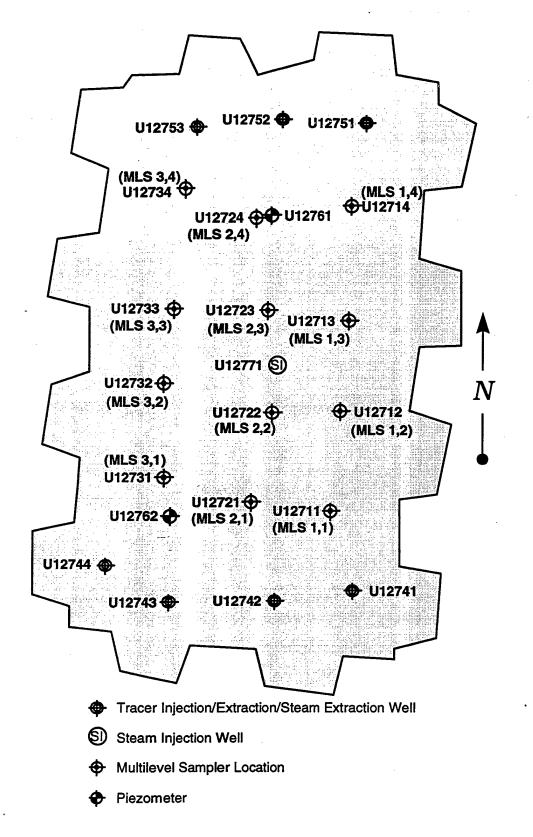


Figure 4. Detailed Layout Showing the Relative Locations of the Injection/Extraction Wells, Piezometers, and MLS Sampling Points.

sampling matrix, allowing the collection of samples from five discrete planer surfaces defined by the 60 individual MLS points (e.g., 12 MLS clusters with five sampling depths per cluster).

## b. Injection/Extraction Wells and Piezometer Construction

The location and spacing of the injection/extraction wells were based on the results of groundwater modeling aimed at optimizing uniform flow conditions within the cell. A bank of four evenly spaced injection wells (U1-2741 through U1-2744) were located along a line approximately 0.3 meter (1 foot) from the south end of the cell. The three extraction wells (U1-2751 through U1-2753) were located along a line offset approximately 0.3 meter (1 foot) from the north end of the cell. A centrally located steam injection well (U1-2771) was installed and used as the primary injection point for the steam.

The injection/extraction wells served dual purposes. During the PITT testing, the injection wells were used to inject the tracers into the cell and maintain a constant hydraulic head at the south end of the cell. The extraction wells were used to extract the cell fluids, again maintaining a constant, but lower, hydraulic head at the north end of the cell. This provided the gradient necessary to maintain flow through the cell during the PITTs.

During the steam injection/vapor extraction phase of the experiment, both the injection and extraction wells served as extraction wells to remove liquid and vapor phase from within the cell while the centrally located injection well was used to inject the steam.

Due to the elevated temperature within the cell during steam injection, all injection/extraction wells were constructed from 2-inch (51 mm) diameter continuous v-wrap stainless steel screen. The screened portions were positioned to span the NAPL smear zone as determined from soil samples taken during cell construction (e.g., approximately 4 to 7 meters [13 to 23 feet] bgs).

Two piezometers were installed on either end of the cell to allow monitoring of the groundwater elevation within the cell. The down-gradient well (north end of cell) was also used to house the extraction pump during both PITTs. These were constructed of schedule 80 PVC,

which was used to allow the proper clearance for the Grundfos Rediflo® pump that was used to maintain flow out of the cell.

Finally, a nested-pair of small diameter (e.g.,  $\frac{3}{4}$ -inch  $\phi$  PVC) monitoring wells were installed just outside the cell on the downgradient (north) end. These were used to monitor groundwater quality during the tracer test to insure that the tracers were not leaking from the cell and also to determine if a vertical gradient existed close to the cell. Table 2 lists the as-built details of all of the wells installed within the cell. Further details covering the well installation methods and procedures are covered in the Phase I Workplan.

TABLE 2. WELL CONSTRUCTION DETAILS

Well ID	Well Type/Description	Material	Total Depth (meters)	Screened Interval (meters)
U1-2741	Injection	2"-	6.95	3.92-6.95
U1-2742	Injection	2"-	7.01	3.96-7.01
U1-2743	Injection	2"-	7.14	4.09-7.14
U1-2744	Injection	2"-φ Stainless Steel	7.30	4.25-7.30
U1-2751	Extraction	2"-	7.28	4.23-7.28
U1-2752	Extraction	2"-	7.21	4.16-7.21
U1-2753	Extraction	2"-	7.16	4.11-7.16
U1-2761	Inside Cell Piezometer/Pumping	2"-φ PVC	7.26	4.21-7.26
U1-2762	Inside Cell Piezometer	2"-\$ PVC	7.51	4.46-7.51
U1-2771	Central Steam Injection	2"-φ Stainless Steel	7.80	4.76-7.80
U1-2781	Shallow Outside Cell Piezometer	³⁄4"-φ PVC	7.20	5.67-7.20
U1-2782	Deep Outside Cell Piezometer	3⁄4"-ф РVС	11.28	9.76-11.28

## B. LEAK TESTING

Once installed and grouted, the cells were leak tested to determine if they leaked and if so, at what rate. This was done by flooding the cells to the top of the NAPL contaminated soil, then observing the water table elevations inside the cell for a period of one week. At the end of that period, the cell was again flooded, recording the volume of water required to bring the water table back up to the top of the NAPL zone. The cell leakage rate was calculated and reported as an average daily rate in liters per day (lpd). The maximum acceptable leakage rate was 0.3 percent of the saturated cell pore volume per day (29 lpd). The leakage rate for Cell 7 was

determined to be approximately 100 lpd, exceeding the acceptable rate. The excess leakage rate was mitigated by maintaining a lower in-cell water level during all demonstration activities than was originally planned (e.g. ≈4.0m vs. 3.6m bgs). This was deemed acceptable to Hill AFB, so no further action was taken.

## C. PRE AND POST TREATMENT CELL CHARACTERIZATION

#### 1. Introduction

Characterizing the spatial distribution of contaminants within the cell before and after treatment was critical for evaluating the effectiveness of the steam injection/vapor extraction technology. The performance of each of the eight innovative technologies will be based primarily on the change in the amount of chemical contamination within each of the test cells. The amount of chemical contamination was determined from the analysis of soil and groundwater samples collected from within the cell both before and after the treatment. The specific compounds selected as target analytes that were used for comparison are listed in Table 3. The results from the PITT tests were also considered in the comparison of the various technologies, although since the PITT is a new technology and not compound specific, these results were not heavily weighted.

## 2. Soil Characterization

Pre- and posttreatment soil samples were collected from a total of 14 locations within the treatment cell. The pretreatment soil cores were collected from eight locations corresponding to the locations of the six injection/extraction wells and two MLS locations. Upon completion of the steam injection treatment, an additional six borings were completed. Figure 5 illustrates the relative locations of these 14 soil borings.

The borings were advanced using a Mobile B-51 auger drill rig outfitted with 4-inch ID, continuous-flight augers. Samples were collected using a 3-inch ID by 4-foot long sampling tube driven by a mechanical drop-hammer at 4-foot intervals as the augers were advanced into the earth. The samples were collected from the NAPL-saturated zone beginning at about 3.6 meters (11.8 feet) below ground surface (bgs) and continuing to 7.3 meters (24 feet) bgs. At one location, the boring extended to the underlying aquitard, approximately 8.2 meters (27 feet) bgs,

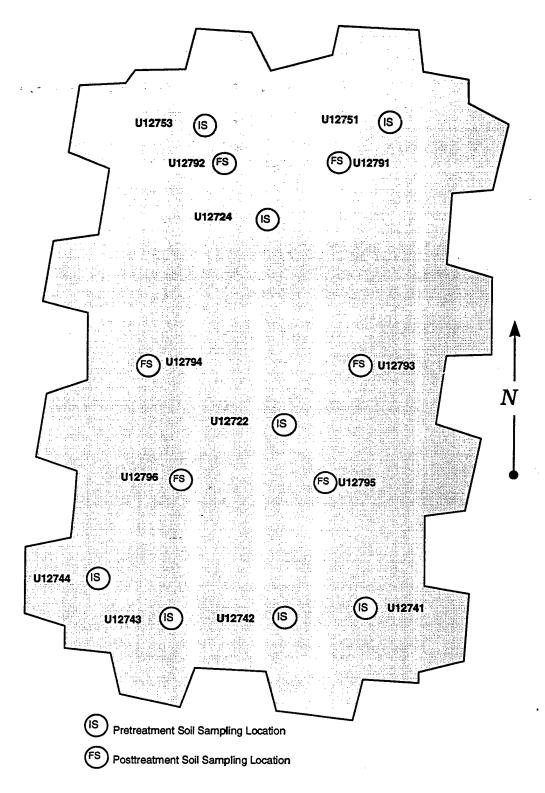


Figure 5. Location of Pre- and Posttreatment Soil Borings.

to confirm its presence. The samples were logged by ARA's field engineer based on the Unified Soils Classification System (USCS). Each sample was screened for total volatile organic compounds (VOCs) using a Photovac MicroTip® photoionization device (PID). The cores were divided into 0.3-meter (1-foot) intervals and sub-samples were collected for chemical analysis.

To minimize the loss of VOCs from the samples during shipping and handling, it was necessary to produce field extracts. The chemical analytical method specified placing approximately 5 grams (0.18 ounce) of soil into a pre-weighed vial containing five-milliliters of dichloromethane, the extractant, and a small amount of hydrochloric acid, a preservative. The samples were capped, agitated to mix thoroughly, then packaged in coolers and shipped to Michigan Technical University (MTU) for analysis by method RSKSOP-72 (Appendix A).

Since an extraction method was used, it was necessary to determine the gravimetric water content of the soil so that the concentrations of the target analytes could be converted from concentrations in dichloromethane to concentrations in soil. This was accomplished by collecting a corresponding sub-sample of soil and analyzing it for soil moisture content as described by ASTM Method D 4959.

A composite sample from each boring was prepared by combining portions from each core section. These composites were analyzed for dioxins, furans, and PCBs by standard EPA methods, using EPA Level III protocol. These samples were collected by Montgomery-Watson personnel and sent to Quantera Laboratories for analysis. These data was sent directly to the EPA and will be incorporated into the master document covering all of the research projects, which will be assembled by the EPA.

#### 3. Groundwater Characterization

The intent of groundwater sampling during the pre- and posttreatment sampling activities was to evaluate the changes in groundwater quality resulting from the steam injection/vapor extraction treatment. These data were also used to determine the effect of the treatment on the individual contaminants partitioning between the soil and groundwater at equilibrium before and after treatment. In addition, these samples were used to estimate the mass of contaminant present in groundwater before and after treatment.

Four separate sampling events were used to achieve the objectives of the groundwater sampling program. There were two under dynamic flow conditions (pre- and posttreatment), and two under static conditions (pre- and posttreatment). Dynamic conditions were defined as the steady-state flow conditions established to conduct the PITT, and samples were collected prior to both the pre- and post-PITT tests. Static conditions were defined as the no-flow conditions within the cell approximately 48 hours following the completion of the pre- and posttreatment. PITTs.

## D. CONCEPTUAL STEAM INJECTION/VAPOR EXTRACTION SYSTEM

In a conceptual cleanup, vertical steam injection wells are placed within the region of contamination and extraction wells are placed within and around this region. Steam can be injected both above and below the water table, assuming contamination exists in both zones. The process is illustrated in Figure 6. The steam injection pressure must be higher than the hydrostatic pressure of the aquifer to enable injection. In the extraction wells, contaminated groundwater and product are removed and the highest practical vacuum is applied. This aids in directing the steam toward the extraction wells. The soil is heated as the steam condenses until it reaches steam temperature, creating a steam zone, which grows toward the extraction wells and pushes much of the contaminant ahead of it. In the steam zone, residual contaminants are volatilized and swept toward the extraction wells by the flowing steam. After steam breaks through in the extraction wells, the injection continues until recovery of contaminants diminishes.

Steam injection can enhance cleanup technologies such as pump-and-treat, soil venting, and bioremediation through several different thermodynamic mechanisms. First, relatively high pressure gradients develop in the steam zone due to the high vapor velocities. These pressure gradients allow the effective displacement of original water and contaminant in place. Liquids that are pushed into the well are pumped until steam breakthrough occurs. Application of a vacuum to the recovery wells during the injection of the steam aids in directing flow toward the well through the vadose zone, and contaminant recovery is identical to that of vapor venting until steam breakthrough. After breakthrough, the steam vapor behaves like air during soil venting,

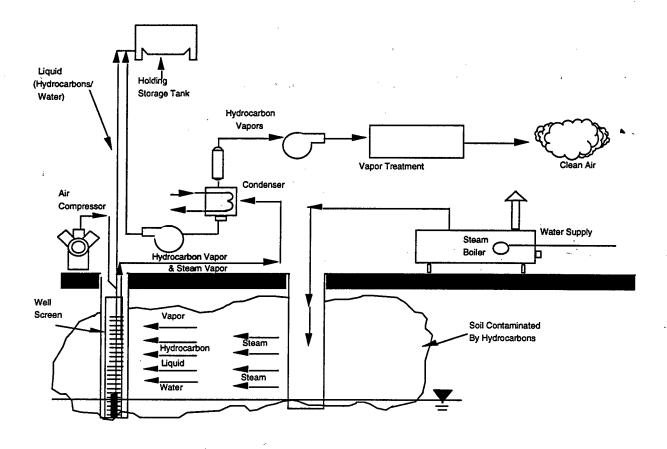


Figure 6. Process Flow Diagram of the Steam Injection/Vapor Extraction System.

except that now the soil is at an elevated temperature. The vapor pressures of typical organic compounds increase by factors from 25 to 40 over those at ambient soil temperatures. This greatly accelerates evaporation rates and reduces remediation duration.

# SECTION III TEST DESCRIPTION

#### A. FLUORESCEIN DYE TRACER TEST

#### 1. Introduction

Before the pretreatment PITT tests, a fluorescein dye tracer test was conducted to establish the sampling plan for the PITT tests, confirm operation of the samplers, and assist in the identification of any preferential flowpaths that might exist within the cell. Although the fluorescein dye does not interact with the NAPL contamination as the partitioning tracers do, it assisted by establishing the time frame for the non-partitioning tracer to flow through the cell. From this test, the travel times for the partitioning tracers were estimated and a sampling schedule determined. The sampling schedule determined during this test served as a baseline to ensure 10 points on each rising edge and 20 points on each falling edge for each partitioning tracer. The schedule remained flexible throughout the PITTs based on the real-time results. One of the goals of the fluorescein dye tracer test was to ensure that significant amounts of oversampling do not occur.

Since the MLS sampling system was used for both the PITT tests and the dye tracer tests, the dye tracer test provided confirmation that all the samplers and the sampling network were operational and fully functioning prior to the PITT. The sampling system details are described under § III.B.2. Figure 4 illustrates the relative locations of the MLS clusters.

The third benefit of performing a dye tracer test was that, combined with the three-dimensional sampling grid, identification of any preferential flowpaths within the cell could be determined from the dye tracer breakthrough curves. For example, if the dye shows up in a downstream point (MLS 1,4) at a depth of 6.1 meters (20 feet) prior to any dye appearing at points MLS 2,4 or MLS 3,4, that would indicate a preferential flow path down the east edge of the cell at a depth of 6.1 meters (20 feet). These types of trends identified during the dye tracer test are beneficial in the analysis of both the PITT results, which showed similar trends, and the steam demonstration in terms of regions that were more likely to be remediated than other regions.

## 2. Test set-up-

The fluorescein tracer test was initiated on April 19th, 1996, and continued for 72 hours. Samples were collected using a variable schedule that collected the first round at the 1-hour point, the second round at the 2-hour mark, and the third round at the 4-hour mark. Sampling continued every 2 hours between 4 and 12 hours and then switched to every 4 hours between 12 and 28 hours. Between 28 and 72 hours, samples were collected every 8 hours. The actual sampling times, as well as the measured fluorescence values of each groundwater sample collected, are included as Appendix B.

Samples were collected from all of the multilevel samplers (MLS) during each sampling round, as well as from the three extraction wells. The sampling of the MLSs was accomplished using the manifold system developed for the PITT testing, as described in § III.B.2.

Fluorescein was selected based on previous experience, nonreactiveness, and documented studies showing fluorescein to be harmless to the environment. The tracer was injected at a concentration of 500 parts per billion (ppb). Based upon a cell size of 3 m by 5 m, 4.3 m of saturated material and a mobile porosity estimate of 17 percent, the saturated pore volume was 9,702 liters (2,563 gallons). The tracer slug was selected to be one-tenth of the saturated pore volume, and, therefore, 969 liters (256 gallons) of tracer were required for the experiment. The initial concentration of the tracer was selected at 500 ppb based upon the detection range of the fluorescence sensor, which covered 50 to 500 ppb. For a 500 ppb mixture in 969 liters (256 gallons) only 0.48 gram (.01 ounce) of the concentrated Fluorescein was required. The water and Fluorescein were thoroughly mixed in a large tank and plumbed into the injection well line for input into the system. The injection and extraction flow rates were each set at 3.8 liters/min (1.0 gallons/min) based upon a desired residence time for both the fluorescence tracer and the PITT tracers of just under 2 days, as well as prior experience from the University of Florida cell experiment.

Once the samples were collected from the MLSs, they were allowed to settle for 2 hours in a dark room before measurement. This allowed any fines in the sample to settle out and not interfere with the measurements. Measurements were made using a 10-AU digital Fluorometer

from Turner Designs. The instrument was calibrated by taking groundwater samples from the site and establishing a blank baseline value. Next, two calibration samples were prepared by mixing groundwater and Fluorescein WT at concentrations of 100 ppb and 400 ppb. The instrument response from these two samples were 87.6 and 397, respectively. These data points were used in an internal calibration routine within the instrument, which allowed the instrument to produce actual concentration readings directly. These concentration values are tabulated in Appendix B.

## B. PARTITIONING INTERWELL TRACER TEST

This section describes the Partitioning Interwell Tracer Tests (PITTs) conducted both before and after the steam injection/vacuum extraction treatment study. The objective of the PITTs was to evaluate the effectiveness of the steam injection by estimating the mass and spatial distribution of NAPL present within the cell both before and after the steam injection treatment. PITT technology was developed in the oil-field industry but has recently been applied to the environmental field by Dr. Gary Pope at The University of Texas, and was first used at Hill AFB at the OU-1 site under the direction of Dr. Mike Annable from the University of Florida.

The PITT test consists of the simultaneous injection of a slug of dilute concentrations of both partitioning and non-partitioning tracers into the test cell under constant flow conditions. The tracer solution is subsequently sampled through the MLS sampling grid and extraction wells. The non-partitioning tracers flow through the cell without reacting with the NAPL, behaving much like a particle of water. The partitioning tracers interact with the NAPL, moving in and out of the NAPL solution at a rate proportional to the NAPL/tracer partitioning coefficient. The net result of this interaction is that the partitioning tracers are retarded with respect to the non-partitioning tracers. By plotting concentration breakthrough curves for each tracer and comparing the first moments (mean residence time) of each, an estimate of the magnitude and distribution of NAPL can be determined.

## 1. Partitioning and Non-Partitioning Tracer Selection

The tracers used for the PITTs were selected based on their partitioning behavior between water and NAPL from the test cell (e.g., the partitioning behavior should emulate the selected

NAPL constituents). In addition, characteristics such as toxicity, degradability, volatility, detectability in the presence of NAPL, cost, and availability were also considered.

The decision as to which tracers to use was based in part on previous work conducted by the University of Florida and the US EPA's RSKRL at the OU-1 site. From this work, a 'short list' of potential tracers was distributed among the researchers. For uniformity's sake, RSKRL requested that each group use at least one nonpartitioning and two partitioning tracers from the 'short list.' The tracers selected for the pretreatment PITT in Cell 7 are tabulated in Table 3.

TABLE 3. TRACERS USED FOR THE PRETREATMENT PITT IN CELL 7.

Tracer	Туре	Partitioning Coefficient*,  K <sub>Nw</sub>	
Bromide	Non-Partitioning	0	
Methanol	Non-Partitioning	<b>≘</b> 0.1	
n-Pentanol	Partitioning	1.4	
n-Hexanol	Partitioning	4.6	
2,2-dimethyl-3-Pentanol	Partitioning	12.9	

<sup>\*</sup>Values from Annable et al., 1994.

A different set of tracers was used for the posttreatment PITT, since the NAPL had changed due to the steam experiment. Methanol proved to be a functional non-partitioning tracer during the pretreatment PITT, so bromide was omitted from the list for the posttreatment PITT. To account for the altered chemical composition and reduced quantity of NAPL as a result of the treatment, and to allow for adequate separation of first moments of the tracer breakthrough curves, it was recommended by Dr. Richard Jackson of Intera, Inc., Austin, Texas, and Dr. Gary Pope from The University of Texas, Austin, Texas, that a tracer with a much higher partitioning coefficient be used for the posttreatment PITT. Based upon their recommendations, the three tracers listed in Table 4 were selected for the post-demo tracer test.

TABLE 4. TRACERS USED FOR THE POSTTREATMENT PITT IN CELL 7.

Tracer	Туре	Partitioning Coefficient*, $K_{Nw}$
Methanol	Non-Partitioning	<b>≅0.1</b>
2,2-dimethyl-3-Pentanol	Partitioning	12.9
n-Heptanol	Partitioning	20.0

<sup>\*</sup>Values from Annable et al., 1994.

## 2. MLS Sampling System

A vacuum sampling system incorporating a centralized vacuum source and distribution system allowed sampling from all 60 MLSs in the system, any combination of isolated rows of MLSs, or a combination of individual MLSs simultaneously. During each sampling event, the sampling apparatus was purged with a minimum of three times the volume of the *upstream* segment, to avoid cross-contamination between sampling events. The upstream segment is defined as consisting of the porous stainless steel filter buried in the ground, the stainless steel transfer line between the filter and the ground surface, and the space within the MLS vacuum manifold between the transfer line and the end of the fill needle that extends into each sample bottle through the lid septum. The vacuum sampling apparatus is shown schematically in Figure 7. Pistons in the cylindrical purge water chambers begin at the bottom of the chamber. When the vacuum pump is switched on, the piston is drawn to the top of the chamber, pulling an ample purge volume through the apparatus and into the purge water chamber to ensure sample quality. After sampling, vials are removed from their guide tubes and the purge water is forced back out of the system for collection in a trough.

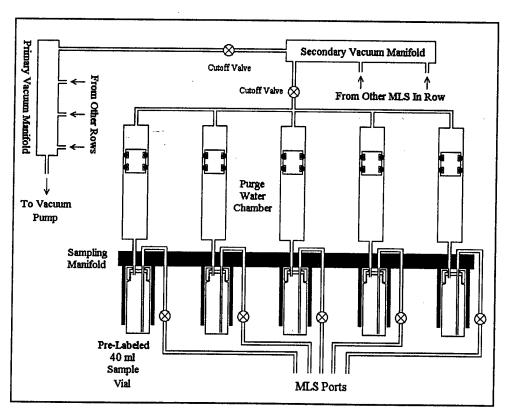


Figure 7. Schematic of Vacuum Sampling Apparatus for MLSs. MLSs to be sampled are selected by operation of cutoff valves.

The purge water was collected in a purge water chamber, which consists of a sealed cylinder within which a piston is installed. The piston is free to move in the direction of the pressure gradient within the cylinder, and requires a differential of about 5 psi to overcome static friction. The piston protects the vacuum pump from drawing in water, and meters the volume of sample drawn from the MLS. The metered volume is greater than the sum of: (a) three volume equivalents of the upstream segment of the sampling apparatus; (b) one sample vial volume, to protect against analyzing the front end of the sample, where reduced air pressure may draw tracer alcohols out of solution, and (c) the volume that the air in the air-filled portion of the upstream segment expands to under the reduced sampling pressure.

Once a sampling round is completed, all filled sample vials were removed from their sampling manifolds and transferred to custody of the analyst(s). Then a positive pressure is applied to the system to discharge the purge water into a trough located below the sampling manifolds. The trough drained into a covered wastewater collection tank at the end of each row.

New sample vials were used for each sampling event. The sample containers were 40-ml certified clean glass vials with Teflon®-faced septum lids. Each vial was labeled with a unique sequential number that was recorded on the master sampling schedule. The master sampling schedule listed all of the MLS locations, and the extraction wells along with the time and date the samples were collected. This provided a quick and efficient methodology for tracking the sampling location and time. After collection, the vials were immediately chilled and stored in a refrigerator at approximately 4°C in Armstrong Laboratory's mobile laboratory. Samples were subsequently subsampled into 2-ml autosampler vials for analysis on a Hewlett-Packard Model 5890 Series II Gas Chromatograph.

## 3. Flow Control System

#### a. Injection System

The water injection control system supplies water to three injection wells simultaneously at a rate necessary to maintain a constant hydraulic head within the cell. This is accomplished by using an active control system that consists of three basic components; a feedback loop, controller circuit, and servo system.

Feedback is accomplished by the use of a sensitive pressure transducer placed in a fixed location at the bottom of one of the injection wells. As the level of the water in the well rises, the voltage output signal of the pressure transducer increases proportionally. This signal is supplied to a controller circuit that produces a corresponding signal based on the difference between the feedback signal and a set point, which coincides with the desired water level. The output signal produced is a function of displacement and velocity. This allows the system to equilibrate at the same water level (displacement), and at any flow rate (velocity). The control signal is fed into a pneumatic servo that produces a pressure proportional to the control signal. The pneumatic pressure, ranging from 20-103 kPa, is connected to a diaphragm actuator that displaces a distance proportional to the pressure in the diaphragm. This mechanism is coupled to a water pressure regulator. The resulting valve assembly provides the ability to regulate water pressure with a control signal. The variable water pressure is fed to a flow control valve that allows adjustment of the flow range. As water pressure is varied by the control signal, the flow rate is varied, maintaining a constant water level within the cell.

The water supply used for both the pre- and posttreatment PITTs was potable water supplied from Hill AFB's potable water system. A 23-cubic-meter (812-cubic-foot) storage tank was used as a reservoir to eliminate the possibility of interrupted flow should the main water supply be shut off temporarily. A water line connected the storage tank to the main feed pump/pressure tank that increased and maintained the water pressure to a usable range of 206-345 kPa. The water was filtered to protect downstream equipment then metered to record the volume of water pumped into the cell. The water meter provided both a digital signal that was recorded by a data acquisition system and an analog gauge for manual recording of the flow.

After the water meter, or totalizer, the water was fed into the pneumatically controlled pressure regulator. This regulated the pressure down to an operating pressure of ≈69 kPa. This allowed room for increased and decreased flow as necessary. The water then flowed through the flow control valve, flow restrictor, and then to a manifold. The manifold fed all three wells by means of a siphon. The siphon connection of all three wells allowed the levels in each well to equalize.

#### b. Extraction System

The water extraction control system removed water from four wells simultaneously at a rate necessary to maintain a constant level at the north end of the cell. This was accomplished by using an active control system similar to the injection system. The control system has three basic components; a feedback system, a controller circuit, and an extraction pumping system.

Feedback was accomplished by the use of a sensitive pressure transducer similar to that used on the injection system. The transducer was placed in a fixed location at the bottom of one well. As the level of the water in the well changed due to pumping, the voltage output signal of the pressure transducer changed proportionally. This signal was amplified and supplied to a controller circuit that produced a control signal based on the difference between the feedback signal and a set point. The set point was the desired water level. The output signal produced was a function of displacement and velocity allowing the system to equilibrate to the same water level at any flow rate. The control signal was fed to a Grundfos® 3-phase motor controller that drives the extraction pump motor at a speed proportional to the control signal, thus controlling the extraction flow rate. The motor speed ranged from 22 Hz to 377 Hz, and although the motor was always running, the flow approached zero, because at 22 Hz there was not enough head produced to pump any water out of the cell.

The extraction water originated in four wells connected together with a siphon. The siphon was used for maintaining a constant and equal water level in the three extraction wells. The pump was placed in a fourth extraction well, and that was also tied into the siphon manifold. The water was pumped through a filter and finally through a water meter. The water meter accumulated the volume of water pumped out as described in the injection system. The water was then directed to a 83-cubic-meter (2,931-cubic-foot) collection tank and later pumped to the base waste treatment facility.

## 4. Alcohol Tracer Chemical Analysis

The analytical methodology for detecting alcohol tracers in groundwater was adapted from the previous experience of Annable et al. (1994) with tracer studies conducted at Operable Unit 1. Analyses for the tracer alcohols were performed by direct injection gas chromatography

using a flame ionization detector (FID). An SOP was developed based on EPA Method 8015 to formalize the method and add pertinent QA/QC topics. The purpose of this SOP was to ensure reliable and reproducible analytical results of the tracer alcohols in groundwater samples for onsite or laboratory-based gas chromatography analyses. The complete SOP is included as Appendix C. The analytes of concern included methanol, n-pentanol, 2,2-dimethyl-3-pentanol, n-hexanol and heptanol.

Approximately 2,000 samples were collected and analyzed during both the pre- and posttreatment PITTs for a total of about 4,000 samples. Two auto-sampler-equipped gas chromatographs (GCs) were employed on site during the tracer tests to keep up with the high volume of samples collected. Both GCs were run continuously but were unable to keep up with the demand. The remaining samples were stored in Armstrong Laboratory's Mobile GC lab refrigerator until the conclusion of the respective PITTs, then shipped to ARA New England Division's laboratory for analysis.

## 5. Computational Analysis of PITT Data

After the chemical analysis of the PITT tracer data was reviewed, the data were forwarded to INTERA Inc., Austin, Texas, for analysis using both the method of moments and the method of inverse modeling techniques. Analysis of the PITT data is based on the chromatographic separation of the partitioning and nonpartitioning tracer responses in the extraction wells and MLSs. The theoretical and experimental foundations for using PITT testing to characterize NAPL are presented elsewhere (Jin et al. 1995; Jin 1995; and Pope et al. 1994; Pope et al. 1995). Below are excerpts from INTERA's data reports discussing the analytical techniques used to process the PITT data.

## a. First Moment Analysis

One simple method of analysis is the method of first moment analysis. The details of the method of first moment theory for the NAPL partition interwell tracer test can be found in Jin et al. (1995) and Pope et al. (1995). Only pertinent equations that relate to the estimation of NAPL volume and average saturation are presented here.

For a partitioning interwell tracer test with multiple injectors and extractors, the volume of NAPL  $(V_i)$  in the swept pore volume of extraction well i, is calculated as:

$$V_{i} = \frac{m_{i}}{M} \frac{(\overline{v}_{p} - \overline{v}_{n})}{K} \tag{1}$$

where M is the total mass of tracer produced,  $m_i$  is the total mass of tracer produced from the extraction well i. K is the partition coefficient of the partitioning tracer defined as the ratio of the tracer concentration in NAPL phase to that in water phase.  $\overline{v}_n$  and  $\overline{v}_p$  are the first moments of the non-partitioning and partitioning tracers, respectively, and obtained by integrating corresponding tracer response curve over the total volume of water injected (v) as:

$$\overline{v} = \frac{\int_0^{V_r} vC_i(v)dv}{\int_0^{V_r} C_i(v)dv} - \frac{V_s}{2},$$
(2)

where  $V_f$  is the total volume of water injected at tracer test cutoff time,  $V_s$  is the total volume of water injected at the end of tracer slug injection and  $C_i(v)$  is the tracer concentration in extraction well i.

The total volume of NAPL  $(V_N)$  is the summation of the volumes estimated from each extraction well and is given by:

$$V_{N} = \sum_{i=1}^{N_{p}} V_{i} \tag{3}$$

where  $N_p$  is the total number of extraction wells.

The retardation factor  $R_f$  is related to the partition coefficient and average NAPL saturation,  $S_N$ , by:

$$R_{f} = 1 + \frac{KS_{N}}{1 - S_{N}} = \frac{\overline{v}_{p}}{\overline{v}_{n}}, \tag{4}$$

The first step in the data analysis process is to evaluate the available field data and select a pair of non-partitioning and partitioning tracers to use for NAPL volume and saturation

estimation. Theoretically, each pair of non-partitioning and partitioning tracers data can give an independent estimate of NAPL volume and saturation. Practically, however, the retardation factor should be greater than 1.2 in order to increase the estimation accuracy (Jin,1995). The retardation factors of pentanol and hexanol from this tracer test are much smaller compared with 2,2 dimethyl, 3-pentanol. Therefore, for the purpose of this experiment, calculations will be based on the tracer response data of methanol and 2,2 dimethyl, 3-pentanol.

To estimate the NAPL volume accurately, the tracer response data should also be complete, because much of the information is contained in the tails of the response curves. It has been shown that the tails of the tracer response curves can be extrapolated with an exponential function (Jin, 1995). The tracer data from this test are complete and can be used without extrapolation. The tracer analysis was done by fitting the tracer response data of methanol and 2,2 dimethyl, 3-pentanol with two smooth curves. NAPL volume estimation is based on the smooth curves. The tracer data from the pretreatment PITT indeed exhibit the exponential decline. Therefore, any incomplete tracer data or the data that showed significant scattering because of the effect of GC detection limits were extrapolated using the exponential decline function to increase the estimation accuracy.

## b. Inverse Modeling Technique

The idea of using the method of inverse modeling for NAPL characterization based on the data obtained from partitioning interwell tracer test was proposed in Jin et al. (1995). Recently, Harneshaug (1997) has successfully used this method to analyze the data from a partitioning tracer test in a similar test cell at the same site conducted by researchers from the University of Florida in 1994 (Pope et al. 1994, Annable et al. 1994). Inverse modeling involves minimizing the difference between the model predictions and observed values by adjusting some unknown model parameters. The most common technique used in inverse modeling is nonlinear least-squares regression.

There are two main steps in analyzing the test cell tracer data using the method of inverse modeling. The hydraulic conductivity distribution in the test cell is first obtained from

the conservative tracer data. This is done by minimizing the differences between field measured tracer concentrations and the model predicted tracer concentrations as:

$$\min f(x) = \frac{1}{2} \sum_{i=1}^{n} (C_{i,model} - C_{i,field})^{2}$$
 (5)

where f(x) is the objective function,  $C_{i,model}$  the model-predicted concentration at location i, and  $C_{i,field}$  the field measured tracer concentration at the same location. The simulator iteratively changes the hydraulic conductivity in each grid-block to obtain the hydraulic conductivity field for which the objective function reaches a minimum. A minimum objective function implies a good match of the field data.

When a good match of the field data was obtained, the corresponding conductivity field was considered as the actual hydraulic conductivity field. The partitioning tracer data were then used to obtain the NAPL saturation distribution. This was done by minimizing the differences between the retardation factors from the field measured data and the retardation factors from the model predicted tracer concentrations. That is,

$$\min f(x) = \frac{1}{2} \sum_{i=1}^{n} (R_{i,model} - R_{i,field})^2$$
 (6)

where  $R_{i,model}$  and  $R_{i,field}$  are the retardation factors calculated from the model predicted data and the field data, respectively.

Similarly, the simulator iteratively changes the NAPL saturation in each grid block to obtain NAPL saturation distribution so that the objective function reaches a minimum. A minimum objective function implies a good match between the model predicted and field measured tracer concentrations. When a good match of the field data was obtained, the corresponding NAPL distribution was considered as the actual NAPL distribution in the test cell.

(1) <u>Numerical Simulators</u>. Two numerical simulators were used for the data analysis. The CONJUGATE code, developed at the University of Texas at Austin (Datta-Gupta, 1992; Kurihara, 1995), and later modified at Texas A&M University by Datta-Gupta (1995), was used to find the hydraulic conductivity distribution. This code uses the conjugate gradient method to minimize the objective function. The other code, UTSTREAM, also developed at the

University of Texas at Austin (Kurihara, 1995, Harneshaug, 1997), was used to estimate the NAPL saturation distribution from the partition tracer data. This code uses Newton's method to minimize the objective function.

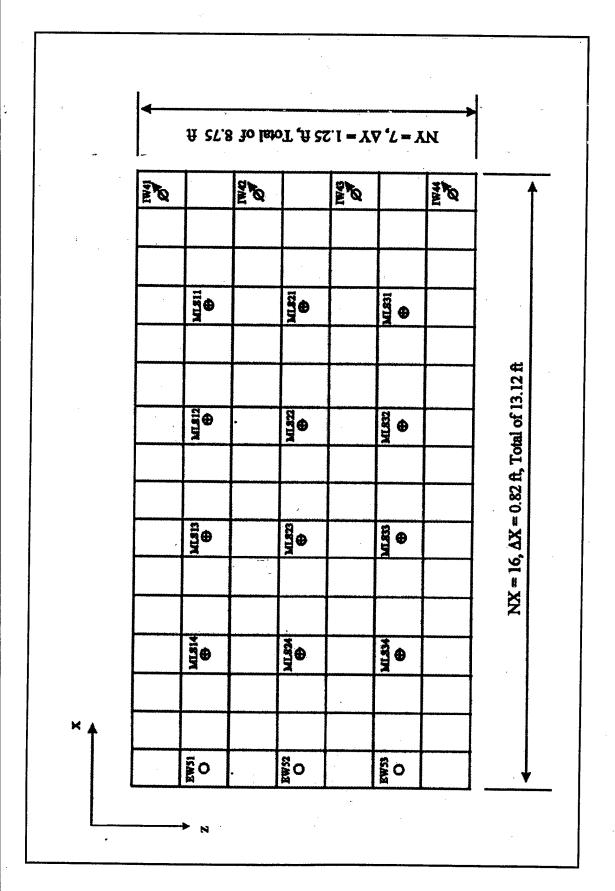
on the well pattern of the test cell. The finite-difference grid used for this simulation has 16 columns (x direction), 7 slices (y direction) and 9 layers (z direction). An x-y plan view of the grid and the well locations is shown in Figure 8, and an x-z cross-sectional view is shown in Figure 9.

Although the main purpose of the inverse modeling simulation was to determine the hydraulic conductivity and NAPL saturation distribution, several forward simulation runs were conducted to estimate the dispersivity and porosity of the sand aquifer within the cell. The results indicate that a dispersivity of  $\cong 0.2$  meter ( $\cong 0.66$  feet) seemed to be suitable. The porosity of the test cell was estimated to be 0.28. The effect of the residual NAPL on the relative permeability to water was assumed to be negligible.

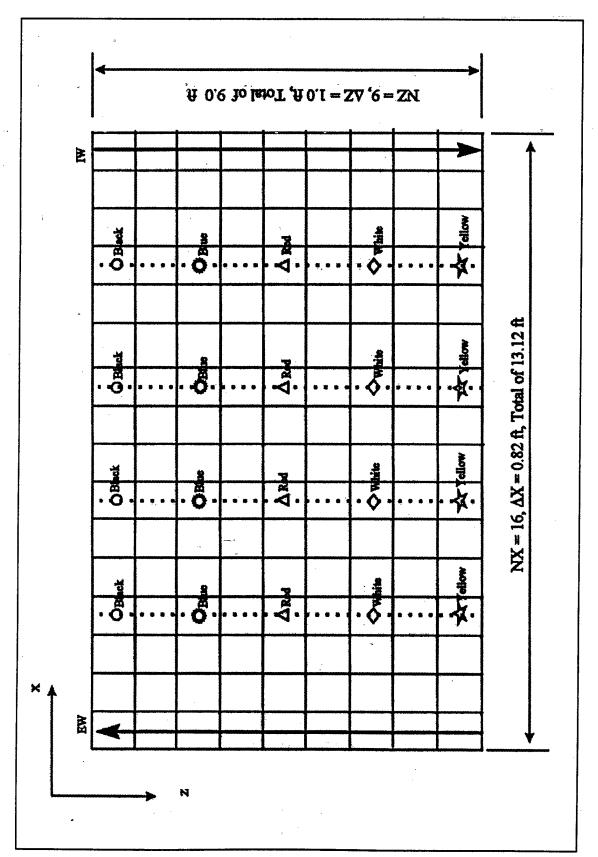
Since the bromide and methanol data are almost identical, either one of them can be used as the conservative tracer. In this report, the methanol data was chosen as the conservative tracer for hydraulic conductivity field estimation, and 2,2-dimethyl, 3-pentanol (partition coefficient of 12.9) was used as the partitioning tracer for the NAPL saturation distribution estimation.

## C. DESCRIPTION OF TREATMENT PROCESS EQUIPMENT

This section describes the equipment installation for the steam injection treatability study. The equipment used in the steam injection study is the equipment trailer; the extraction, injection, and monitoring wells; and the piping to the wells. The equipment trailer contained a steam generator, a condenser, a NAPL/water separator, a water cooling system, a regenerative blower and an activated carbon adsorption system. This system is illustrated in the process flow diagram, shown previously in Figure 6. The subsurface installation of wells, piezometers, and monitoring and sampling probes was described previously in § II.A.2.



Plan View of Simulation Grid and Locations of Injection (IW) and Extraction (EW) Wells and Multilevel Samplers (MLS). Figure 8.



Cross-sectional View of the Simulation Grid and Multilevel Sampler (MLS) Location. Figure 9.

#### 1. Liquid Extraction and Treatment System

Positive displacement pneumatic pumps were used to pump liquids from the four extraction wells located nearest the corners of the test cell (Wells U1-2741, U1-2744, U1-2751, and U1-2753 shown in Figure 4). The liquids were pumped into the bottom of the condenser located on the process trailer and then through a NAPL/water separator. Separated NAPL was routed through a double-contained pipeline to a tank in the effluent tank farm. Contaminated water from the NAPL/water separator was cooled in a heat exchanger. Cooling water for the heat exchanger was provided by a water loop that passed through a small cooling tower located on the process trailer. The cold, contaminated water was sent to the condenser to cool the extracted vapors. When the water level in the condenser reached a pre-set height, contaminated water was automatically pumped through a double-contained line to a storage tank in the tank farm. The volume of liquid in the process equipment was held to a tight tolerance such that the discharge rate to the storage tanks was equal to the extraction rate from the test cell.

## 2. Vapor Extraction and Treatment System

The vapor control and treatment system consisted of a condenser, a regenerative blower, and a carbon adsorption vessel. The equipment was located on the process trailer and connected to the extraction wellheads through a manifold and piping. The regenerative blower supplied a vacuum for the extraction of vapors from the four extraction wells. The vapors passed through a condenser to remove steam and cool the noncondensable gases. Condensed liquids were pumped directly into the NAPL/water separator as described above. After the condenser, the vapors passed into the blower and were pumped through a 180-kilogram (397-pound) vessel of granular activated carbon before release to the atmosphere.

#### 3. Steam Generation System

Steam was provided by a small, skid-mounted electric boiler, located on the process trailer. The steam was carried to the single injection well (U1-2771) at the center of the test cell through insulated steel piping.

## 4. Subsurface Temperature Monitoring System

Thermocouples were installed into the subsurface with each of the multilevel samplers shown in Figure 4. In ten of the MLSs, strands with eight thermocouples spaced 0.8 meter (2.6 feet) apart were installed. In MLSs U1-2721 and U1-2722, the thermocouples were spaced at 0.3-meter (1-foot) intervals for a total of 15 per strand. The thermocouples extended from roughly 7 meters (23 feet) below the ground surface to 1.5 meters (5 feet) below the ground surface. The temperatures at a location were logged with a computerized data acquisition system accepting up to 15 thermocouples. After logging, the system was manually disconnected and moved to a new MLS location. Temperatures were recorded regularly throughout the study.

#### 5. Instrumentation and Control

The steam injection wellhead had pressure and temperature gauges, an isolation valve, and a pressure relief valve. Extraction wellhead instrumentation and control were provided for both the vapor and liquid flow streams. The system consisted of pressure and temperature gauges, flow meters, manually operated flow control valves, air-operated submersible pump flow controllers and sampling connections. The multilevel samplers were monitored with pressure gauges and thermocouple connections. Control of the soil vapor extraction system included suction and discharge pressure gauges, a vapor flow meter, a discharge temperature monitor, manually operated flow control valves, and an automatic vacuum relief valve.

Automatic control was provided to protect equipment and personnel in emergencies caused by equipment failure. The first level of automatic system control and protection was a local emergency shutdown switch of all facility equipment. The second level of control was the protection of equipment by automatically monitoring the water level in the condenser. This allowed excess water to be discharged as appropriate and allowed make-up water to enter the system as needed.

#### D. PHASES OF IMPLEMENTATION

The subsections that follow describe the three major phases of the treatability study. The phases are summarized in Table 5. The first phase consisted of dewatering the test cell and performing soil vapor extraction (SVE) to provide a baseline for comparing the recovery

TABLE 5. SUMMARY OF OPERATIONAL PHASES.

PHASE	ACTIVITY	DURATION
Groundwater and Soil		
Vapor Extraction	De-Watering of the Test Cell	7 hours
	SVE with Injection Well Closed	12 hours
ar An grap	System Shutdown for Rebound	9 hours
	SVE with Injection Well Open to Atmosphere	4 hours
·	System Shutdown for Rebound	2 hours
	SVE with Injection Well Open to Atmosphere	10 hours
	System Shutdown for Rebound	16 hours
	SVE with Injection Well Closed	21 hours
Steam Injection		
and Dual-Phase Extraction	Steam Injection for Initial Cell Heating	19 hours
	Steady-State Steam Injection and Extraction	- 54 hours
	Steam Injection with Reduced Extraction	22 hours
·	Steady-State Steam Injection and Extraction	4 hours
Soil Vapor Extraction		
and Cell Cooling	SVE with Injection Well Open to Atmosphere	357 hours
(No Steam Injection)	Re-Hydration of the Test Cell	5 hours

enhancement from steam injection. The second phase was steam injection with dual phase extraction. The third phase cooled the test cell by continuing SVE after ceasing steam injection. The results of each test phase are presented and discussed in subsequent sections.

## 1. Groundwater and Soil Vapor Extraction

In the first phase of the study, the test cell was dewatered and a variety of SVE tests were performed. Due to the elevated water level required for the PITT, a total of 1,302 liters (344 gallons) were removed in 6.5 hours to dewater the test cell. NAPL was not visible in the extracted water during this initial pumping. The water level was lowered 1.23 meters (4 feet) during the dewatering and the area of the test cell is approximately 14 meters squared (150 feet squared). For these dimensions, the air-filled porosity of the dewatered soil was about 7.5 percent.

After the dewatering was complete, a series of soil vapor extraction tests were performed over a 7-day period. Chemical and physical data were collected during each test. Physical data from the tests were used to determine the air permeability of the dewatered soil. Chemical data were collected to determine the equilibrium vapor concentrations of volatile organic compounds (VOCs) in the cell, to predict long-term SVE performance for comparison with heating, and to evaluate mass transfer constraints. As shown in Table 5, SVE was performed with the center injection well either open or closed, and short shutdown periods were implemented to assess concentration rebound.

## 2. Steam Injection and Dual-Phase Extraction

In the second phase, steam was injected in the center well while vapors and liquids were pumped from four extraction wells. Throughout the steaming period, the injection rate was maintained at a steady 113 kilograms (250 pounds) per hour. Temperature profiles were recorded at each MLS to track the growth of the steam zone. Steam reached Extraction Well U1-2741 after 11 hours of injection and well U1-2744 after 14 hours. The steam zone reached wells U1-2751 and U1-2753 after 19 hours of injection. Shortly after steam breakthrough in all four extraction wells, the flow became effectively steady with the steam extracted equaling the steam injected. This steady operation was continued for 54 hours. The steady flow was followed by variations in the extraction rate from different wells and observation of the resulting changes in the steam zone growth. These tests lasted 22 hours. The period of variable extraction was followed by steady flow and extraction for 4 hours before the steam injection was terminated.

A total of 2,540 liters (671 gallons) of water were removed in the form of steam condensation and groundwater during this phase. NAPL was removed, but only about 8 liters (2 gallons) were separated by the system. The low yield of NAPL is a result of one or all of the following: the NAPL was not significantly mobilized, the main process pump emulsified the NAPL in the water such that separation did not occur, or the estimate of NAPL in the cell from the tracer test was too high. Extensive chemical and physical data were collected during this phase.

## 3. Soil Vapor Extraction and Cell Cooling

In the third phase, only soil vapor was extracted. The injection well was opened to the atmosphere to allow air to be drawn through this pathway. The thermocouples were monitored closely following the cessation of steam to observe the cooling rate in the cell. This phase lasted for 15 days. Water was extracted in vapor form, and a total of 3,320 liters (877 gallons) were removed. Chemical and physical data were collected at a reduced frequency as compared to the steam injection phase. At the conclusion of the test, the cell was re-hydrated back to its original water level. A total of 2,220 liters (587 gallons) were injected.

## 4. Sampling and Analyses for Process Performance

The number of samples collected for chemical analyses are summarized in Table 6. Eighty-one process vapor samples were collected during the test and analyzed with the on-site gas chromatograph (GC). Nine vapor samples were collected and sent off-site for analysis by a GC and mass spectrometer (GC/MS). Forty-one liquid effluent samples from the process were collected and analyzed with the on-site GC. Twenty-five liquid effluent samples were sent to an off-site laboratory for analysis with a GC/MS. The off-site analyses were used to verify and calibrate the on-site analytical work.

TABLE 6. NUMBER OF SAMPLES COLLECTED FOR CHEMICAL ANALYSIS.

		Number of Samples					
Sample Matrix Analysis Method	Analysis Level	Primary	Duplicate	Ambient Blank	Trip Blank	Equipment Blank	Total
Soil Vapor			100			3	
VOCs (TO 14)	III	9	0	0	0	0	9
VOCs Onsite GC	П	81	0	9	0	8	98
TPH	III	9	0	0	0	0	9
Liquid						130 130 130 130 130 130 130 130 130 130	
VOCs (SW 8240)	Ш	25	0	0	0	0	25
VOCs (Onsite GC)	II	41	1	0	0	5	47
SVOCs (SW8270)	Ш	0	0	0	0	0	0
TPH (SW8015)	III	0	0	0	0	Ö	Ô
Sediment						.£3	
VOCs (SW 8240)	Ш	0	0	0	0	0	0
SVOCs (SW8270)	III	0	0	0	Ō	ő	Õ
TPH (8015)	Ш	0	0	0	0	ő	Õ

## SECTION IV RESULTS

## A. FLUORESCEIN DYE TRACER TEST

Responses from each of the samples collected during the dye tracer experiment were tabulated and plotted. Both the tabulated results and the individual time histories from each of the MLS sampling points are presented in Appendix B. For the following discussion, we will refer to the individual time histories as Figures B-1 through B-15. Analysis of the tabulated results indicates that only 5 of the 60 MLS samplers were non-productive during some portion and these were all corrected during the course of the dye tracer test or before the PITT test. The fluorescent responses for each MLS are plotted in a time history format for each MLS such that the dye tracer break through can be observed.

#### 1. Extraction well results

Using a flow rate of 3.8 liters per minute (lpm) (1 gallon per minute) as described in the test set-up, the initial front of the dye tracer took approximately 5 hours to reach the center extraction well as shown in Figure B-14. The peak of the dye tracer concentration reached the center extraction well at the 10 hour sampling interval. It is interesting that both of the side extraction wells show lower concentrations and a slower response. This is most likely due to the reduced radius of influence of the these wells due to boundary effects of the cell walls, but could also be due to preferential flow paths. Depth variations as presented for the MLSs are not possible in the extraction wells since they were screened over the entire saturated thickness.

#### 2. Row 1 - MLSs

Results from the fluorescent testing of the samples collected from the row closest to the injection point (Figures B-1 through B-3) show a very strong rise in concentrations, initially with a peak between 6 and 14 hours for nearly all samplers located in this row. Nearly all of the tracer slug has passed this row by the 20-hour mark. Both MLS 1,1 and MLS 3,1 show curves exactly as expected. From these two locations it is interesting to note that the concentrations are highest at both the shallowest (4.3 meters, black [14 feet]) depth and the deepest (6.7 meters, yellow [22]).

feet]) depth, with all responses peaking above 100 ppb. The measurements at the center MLS in this row (MLS U1-272,1) show a different response trend, with only the lower two samplers barely reaching the 100-ppb level and the upper samplers showing a much different and slower breakthrough. This indicates that the region in the center of the cell near the top of the saturated region is less permeable than the side and lower portions of the cell.

#### 3. **Row 2 - MLSs**

All three of the deepest samplers (6.7 meters [22 feet], yellow) located on row 2 indicate a rapid flow of the tracer past this row as shown in Figures B-4 through B-6. In all three instances the concentrations peak at around 3 to 5 hours and are completely diminished by 20 hours. The samplers located at the 6-meter (20-foot) depth (white) show a fairly similar response at MLS 1,2 and MLS U1-272,2 but the response is delayed at MLS 3,2. The upper most samplers also show good peak breakthrough response at locations MLS1,2 and MLS U1-272,2, but with a small amount of retardation. Difficulties were encountered with the sampling manifold for this row, making sample collection difficult for some depths. The samplers in the middle region (4.9 and 5.5 meters [16 and 18 feet] deep; blue and red, respectively) do not show much breakthrough response, but rather a slow increase in the fluorescence concentration.

#### 4. Row 3 - MLSs

Results from the samplers in the third row are similar to those in the second row. Early breakthrough of the tracer is noted at the 6.7-meter (22-foot) depth (yellow), especially on the sides and slightly less in the center (Figures B-7 through B-9). On the west side of the cell (MLS U1-272,3 and MLS 3,3) response is also noted in the 10- to 20-hour time frame for a peak response at a depth of 6 meters (20 feet) (white). Breakthrough at this depth on the other side of the cell (MLS 1,3) is significantly delayed and the peak does not occur until 30 to 40 hours. Also at the same location, the sampler at 5.5 meters (18 feet) (red) exhibits this same response. At location MLS 3,3 the breakthrough at a depth of 4.9 meters (16 feet) (blue) is very similar to the breakthrough at 6 meters (20 feet) (white), although the flow at the 4.3-meter (14-foot) depth (black) is retarded significantly. The breakthrough of the fluorescent dye in the sampler located at the 4.3-meter (14 foot) depth (black) for this row is delayed more than at other rows such as 2 and 4.

#### 5. Row 4 - MLSs

The last row of samplers before the extraction wells again shows many of the same trends noticed in the early rows. The earliest breakthrough occurs at the 6.1- (white) and 6.7-meter (20-and 22-foot) (yellow) depths at all three locations as shown in Figures B-10 through B-12. There is breakthrough, although significantly retarded relative to the flow at deeper depths for the samplers located at depths of 4.9 meters (16 feet) (blue) and 5.5 meters (18 feet) (red). This response is especially true along the sides. The upper most sampler level (4.3 meters [14 feet], black) shows a fluorescent peak at the 40-hour time frame, indicating that flow through the upper materials is significantly slower than the lower materials but not nearly as slow as the 4.9- to 5.5-meter (16- to 18-foot) region.

Overall the fluorescent tracer test proved beneficial and provided excellent results for determining an appropriate sampling schedule for the PITT. It also provided some insight regarding possible preferential flow paths. Based upon the tracer breakthrough results, we expected the non-partitioning tracer to take approximately 4 to 6 hours to reach the extraction wells and the peak to occur at approximately 10 to 14 hours. To reach the extraction well this quickly, the tracer is flowing through the lower materials at a depth of 6.1 meters (20 feet) to 7.3 meters (24 feet) bgs. This is the most permeable region of the cell. Another trend indicated by the fluorescent tracer test is that flow that occurs through the middle of the sampled region (i.e., depths of 4.9 meters [16 feet] and 5.5 meters [18 feet]; blue and red, respectively) is retarded when compared to the both the 6.1-meter (20-foot) (white) and 6.7-meter (22 foot) (yellow) depths, as well as the 4.3-meter (14-foot) (black) depth. Analysis of a CPT penetration profile from a test conducted approximately 23 meters (75 feet) away indicates that the soil materials at the 4.3-meter (14-foot) (black) to 4.9-meter (16-foot) (blue) depth may contain more fines. Figure 10 presents this CPT profile and shows that the tip stress is reduced from depths of 4.3 meters (14 feet) (black) to 4.9 meters (16 feet) (blue). Unfortunately, the sleeve response in this region is negative, so the friction ratio can not be calculated, but it is postulated from material changes seen in the tip stress response and from other CPT profiles in the area that the finegrained content increases slightly in this region, resulting in a less permeable region.

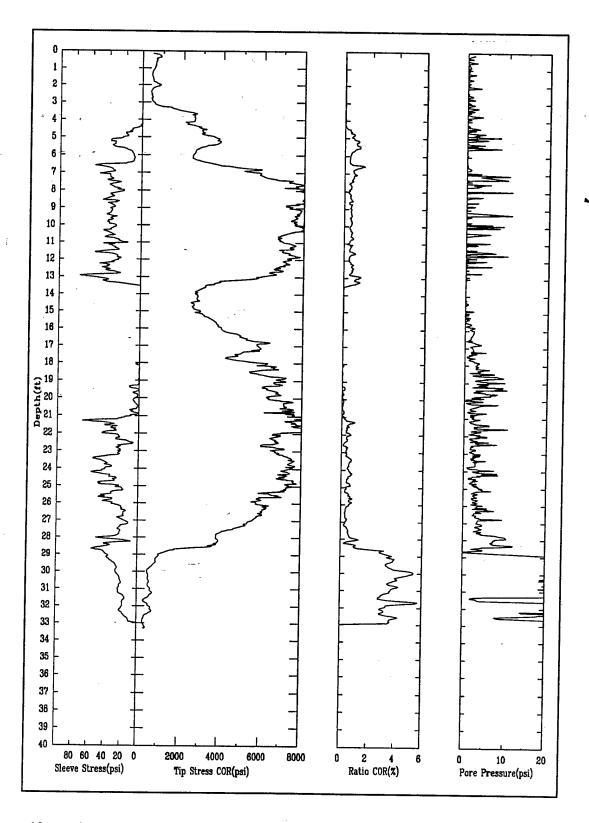


Figure 10. CPT Profile Illustrating Reduced Tip Stress from Depths Ranging from 4.0 to 4.9 Meters (13 to 16 feet) at a Location Approximately 23 Meters (75.5 feet) South of Cell 7.

Another note is that generally the breakthrough occurred earlier at the edge sampler locations than in the center region of the cell. This could possibly be due to disturbance effects caused by the vibratory installation of the sheet pile walls of the cell.

## B. PARTITIONING INTERWELL TRACER TEST

#### 1. Pretreatment

#### a. Method of Moments

The NAPL volume and saturation were estimated as a function of tracer cutoff time. As expected, the estimated NAPL volume and saturation approach a plateau as the tracer test approaches completion. The NAPL volume was estimated by first calculating the first moment of each tracer response curve using Equation (2) in § III.B.5.a. Equation (1) was then used to estimate the volume of NAPL in each swept volume, and Equation (4) was used to estimate the NAPL saturation.

Figure 11 shows the tracer response of methanol and 2-2 dimethyl, 3-pentanol in extraction Well U1-2751 and their corresponding smooth fitting curves. Figure 12 shows that about 27 percent of the total methanol and 23 percent of the total 2-2 dimethyl,3-pentanol injected were recovered from the extraction Well U1-2751 during the tracer test. Figure 13 indicates that tracers captured by extraction Well U1-2751 swept a pore volume of approximately 3.6 cubic meters (127 cubic feet). Figure 14 indicates that the average NAPL saturation in this swept volume is about 5.8 percent, which corresponds to a total volume of approximately 208 liters (55 gallons) as shown in Figure 15.

The tracer responses of methanol and 2-2 dimethyl, 3-pentanol in the extraction Well U1-2752 and their smooth fitting curves are shown in Figure 16. Approximately 29 percent of the total methanol and the total 2-2 dimethyl,3-pentanol injected were recovered from extraction Well U1-2752 as shown in Figure 17. The estimated capture zone of extraction Well U1-2752 is about 2.2 m³, as shown in Figure 18. Figure 19 indicates that the average NAPL saturation in this swept volume is about 5 percent, which corresponds to a total volume of 106 liters (28 gallons) as shown in Figure 20.

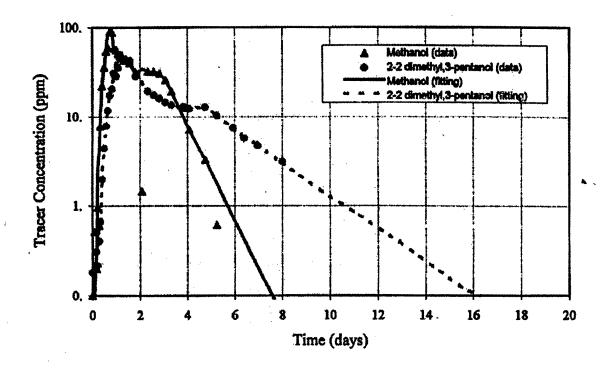


Figure 11. Extraction Well U1-2751 Tracer Response Data and Corresponding Fitting Curves.

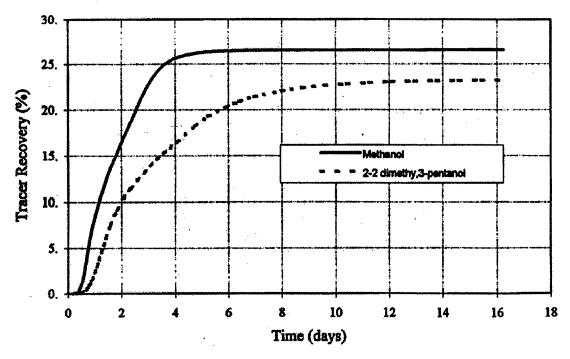


Figure 12. Extraction Well U1-2751 Tracer Recovery.

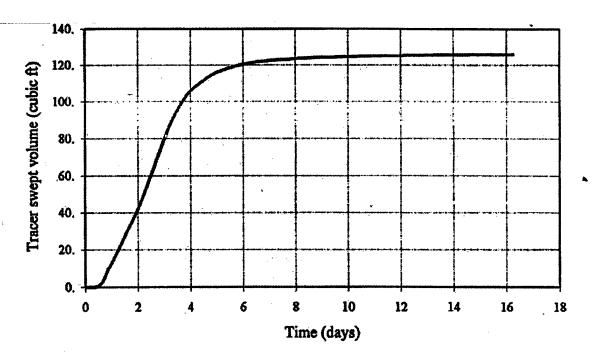


Figure 13. Pore Volume Swept by Tracers Captured by Extraction Well U1-2751.

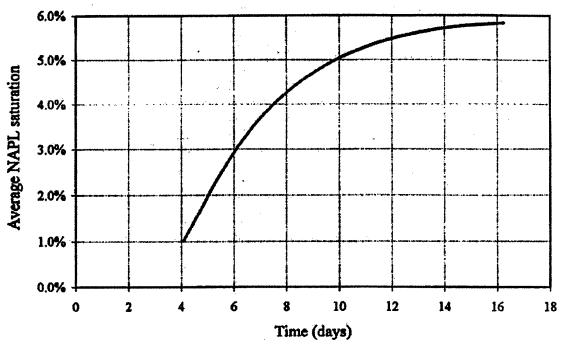


Figure 14. Estimated Average NAPL Saturation in the Swept Volume of Extraction Well U1-2751.

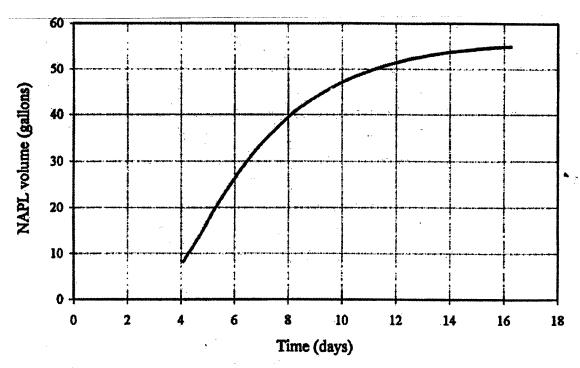


Figure 15. Estimated NAPL Volume in the Swept Volume of Extraction Well U1-2751.

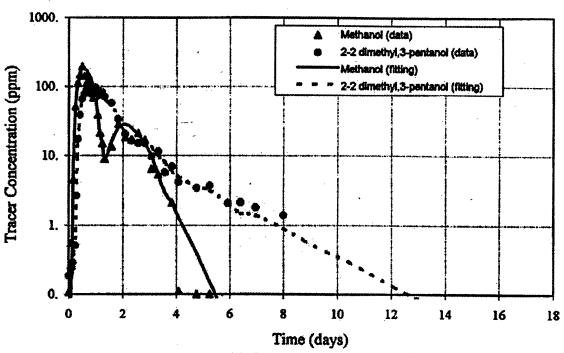


Figure 16. Extraction Well U1-2752 Tracer Response Data and Corresponding Fitting Curves.

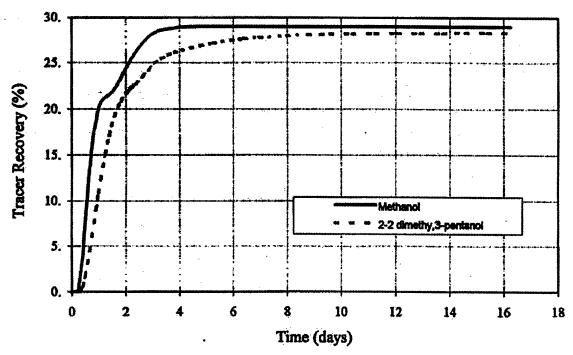


Figure 17. Extraction Well U1-2752 Tracer Recovery.

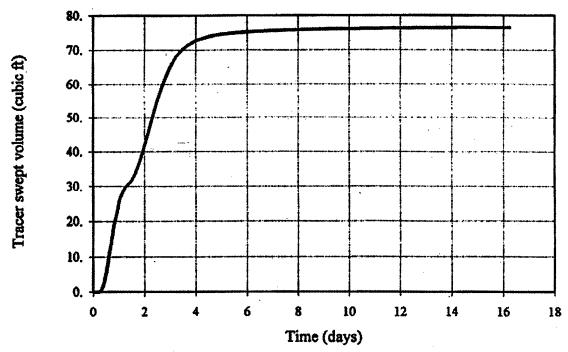


Figure 18. Pore Volume Swept by Tracers Captured by Extraction Well U1-2752.

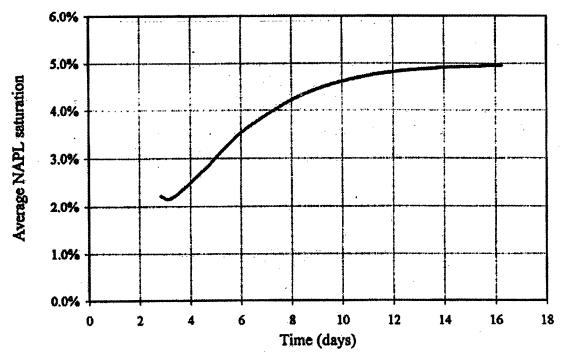


Figure 19. Estimated Average NAPL Saturation in the Swept Volume of Extraction Well U1-2752.

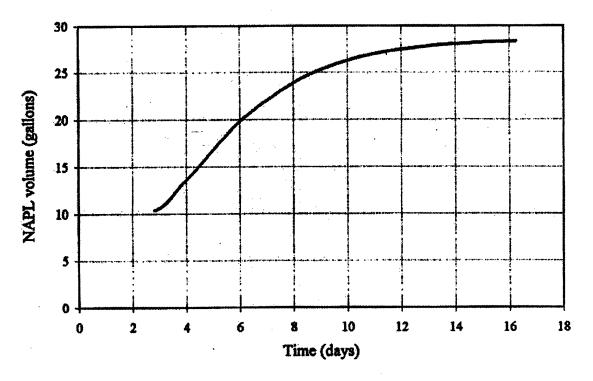


Figure 20. Estimated NAPL Volume in the Swept Volume of Extraction Well U1-2752.

Figure 21 shows the tracer response of methanol and 2-2 dimethyl, 3-pentanol in Extraction Well U1-2753 and their corresponding smooth fitting curves. Figure 22 shows that about 27 percent of the total methanol and 23 percent of the total 2-2 dimethyl,3-pentanol were recovered from extraction Well U1-2753. Figure 23 indicates that the tracers captured by Extraction Well U1-2753 swept a pore volume of 3.6 m<sup>3</sup>. Figure 24 indicates that the average NAPL saturation in this swept volume is about 4.3 percent, which corresponds to a total volume of approximately 155 liters (41 gallons), as shown in Figure 25.

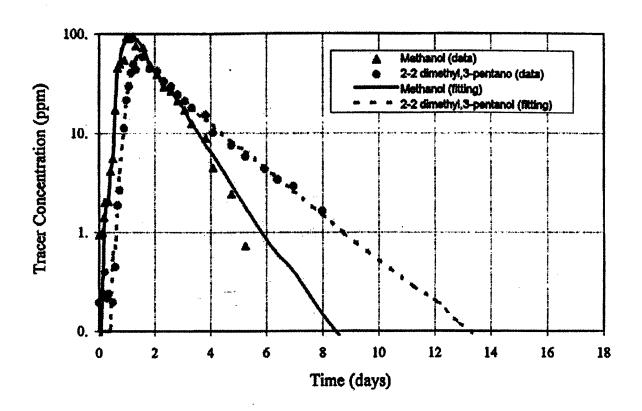


Figure 21. Extraction Well U1-2753 Tracer Response Data and Corresponding Fitting Curves.

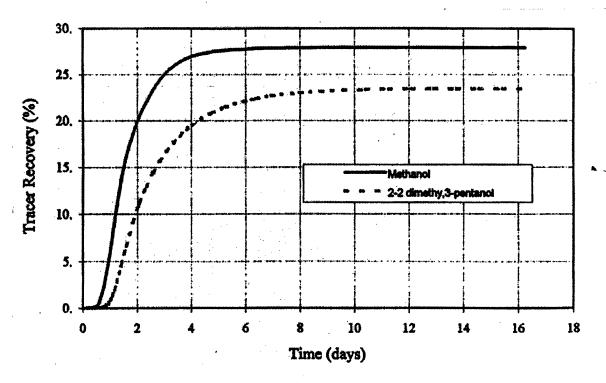


Figure 22. Extraction Well U1-2753 Tracer Recovery.

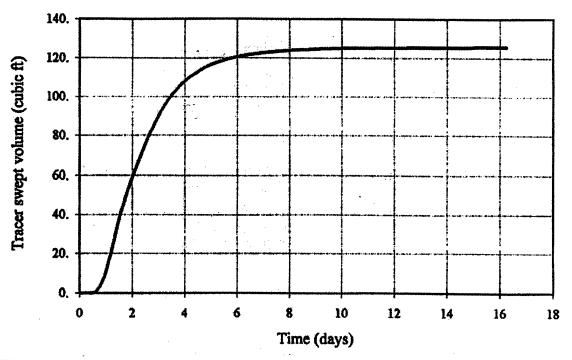


Figure 23. Pore Volume Swept by Tracers Captured by Extraction Well U1-2753.

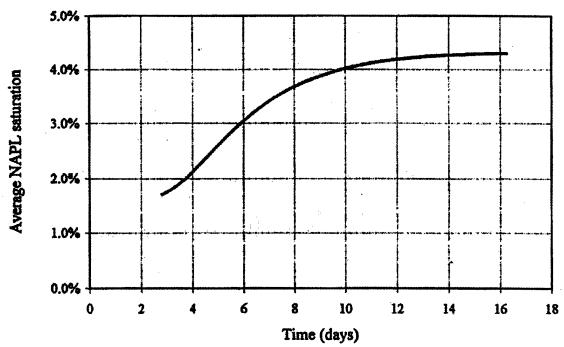


Figure 24. Estimated Average NAPL Saturation in the Swept Volume of Extraction Well U1-2753.

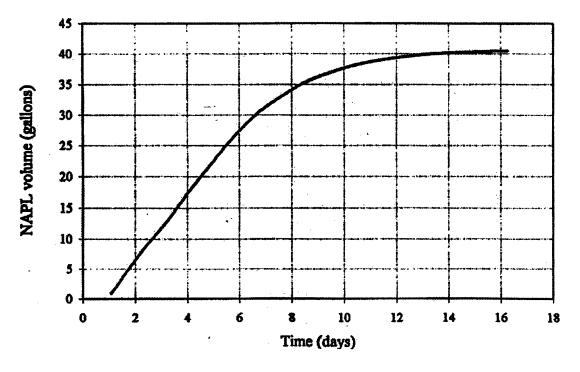


Figure 25. Estimated NAPL Volume in the Swept Volume of Extraction Well U1-2753.

The total volumes of NAPL in the saturated zone of the test cell are 469 liters (124 gallons), which are the summation of the NAPL in the swept volumes of three extraction wells estimated using Equation (3). The recoveries of the methanol and 2,2-dimethyl-3-pentanol tracers are summarized below in Table 7.

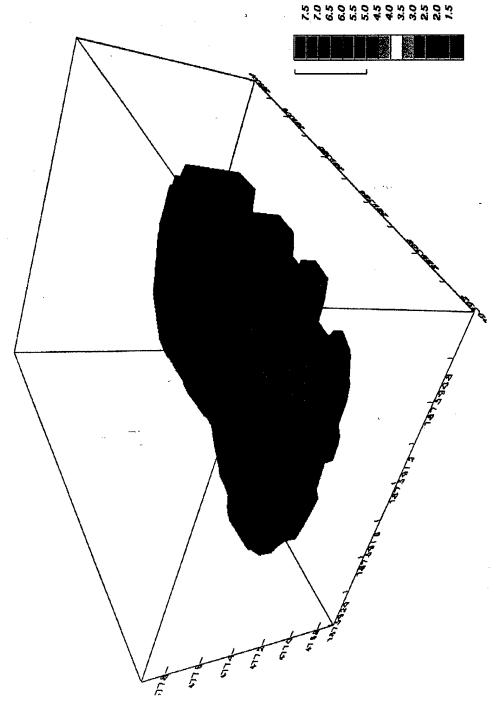
TABLE 7. RECOVERIES OF PARTITIONING AND NON-PARTITIONING TRACER ALCOHOLS.

Extraction Well ID	Methanol Recovery (Non-Partitioning)	2,2-Dimethyl-3-Pentanol Recovery (Partitioning)
51	27%	23%
52	29%	29%
53	27%	23%
	Total = 83%	Total = 75%

The results indicate that only 83 percent of the methanol and 75 percent of the 2,2-dimethyl-3-pentanol were recovered from the extraction wells. This could be attributed to a variety of reasons, including evaporation of the alcohols from the tank prior to injection and a small amount of residual tracer mix that remained in the injection tank. There was also a significant mass of tracer removed from the MLS points due to the quantity of samples collected throughout the duration of the test.

Figures in Appendix E illustrate the tracer responses of methanol and 2-2 dimethyl-3-pentanol, estimated retardation factor and residual NAPL saturation at each of the multilevel sampler (MLS) points except MLS32 black, MLS13 red and yellow, for which the tracer data were not available. Table 8 summarizes the estimated NAPL saturation results based on these monitor point tracer data. It should be noted, however, that the estimated residual saturation does not necessarily indicate the residual saturation at the monitor point, rather, it is the average NAPL saturation in the stream tube connecting the injection well and the monitor point.

Based on the estimates of the NAPL saturation derived from the pretreatment PITT, a three-dimensional "potato in space" was developed using EarthVision®, illustrating the distribution of NAPL in the cell (Figure 26).



Distribution of NAPL Determined from the Method of Moments Analysis of the Pretreatment PITT Data in Percent Saturation. Figure 26.

TABLE 8. SUMMARY OF THE ESTIMATED NAPL SATURATION BASED ON THE PRETREATMENT PITT.

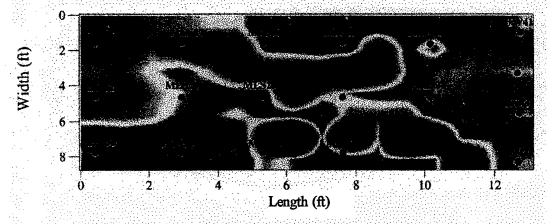
Saturation ( percent)	Black (4.3 m, 14 ft)	Blue Red (4.9 m, 16 ft) (5.5 m, 18 ft)		White (6.1 m, 20 ft)	Yellow (6.7 m, 22 ft)	
MLS11	2.8	1.4	6.3	6.5	4.2	
MLS21	<b>2.3</b> -	5.6	7.9	3.4	6.0	
MLS31	3.0	3.8	6.8	6.2	3.3	
MLS12	3.0	5.3	2.8	5.7	4.6	
MLS22	2.5	3.3	5.9	5.7	4.4	
MLS32	N/A	5.7	6.1	8.0	5.0	
MLS13	2.9	4.7	N/A	5.6	N/A	
MLS23	2.6	3.2	5.6	6.5	4.2	
MLS33	2.8	4.4	5.3	5.6	4.1	
MLS14	3.5	3.7	5.3	6.7	4.5	
MLS24	3.0	3.1	6.2	6.4	2.3	
MLS34	3.1	3.5	5.8	6.9	4.6	

### b. Inverse Modeling Technique

In the conductivity distribution estimation run, a hydraulic conductivity field that roughly resembles the hydraulic conductivity based on the first moment analysis of methanol was used as the first approximation. Then the CONJUGATE simulator was run to optimize the hydraulic conductivity field to improve the matches of the field methanol data. Cross-sectional and plan views of the resulting hydraulic conductivity field are shown in Figure 27. This hydraulic conductivity field has an arithmetic average of  $2.7 \times 10^{-2}$  cm/s. This is typical for gravels, which range from  $10^{-1}$  to  $10^{2}$  cm/s. Figure 28 shows the histogram of the resulting hydraulic conductivity field. This hydraulic conductivity field was obtained after 35 iterations with the CONJUGATE simulator. Each iteration requires the forward problem to be solved 1008 times (once for each gridblock). The objective function, the sum of the least squares differences between simulated tracer concentrations and field methanol concentrations, was reduced from 5.6 to 2.1.

Upon completing the hydraulic conductivity field matching run, UTSTREAM was run to find the NAPL saturation distribution. A uniformed residual NAPL saturation of 5 percent

# a) A plan view at YELLOW multilevel sampling points.



# b) A cross-sectional view along the extraction well EW51.

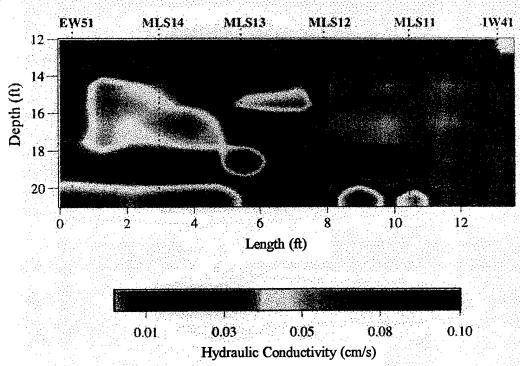
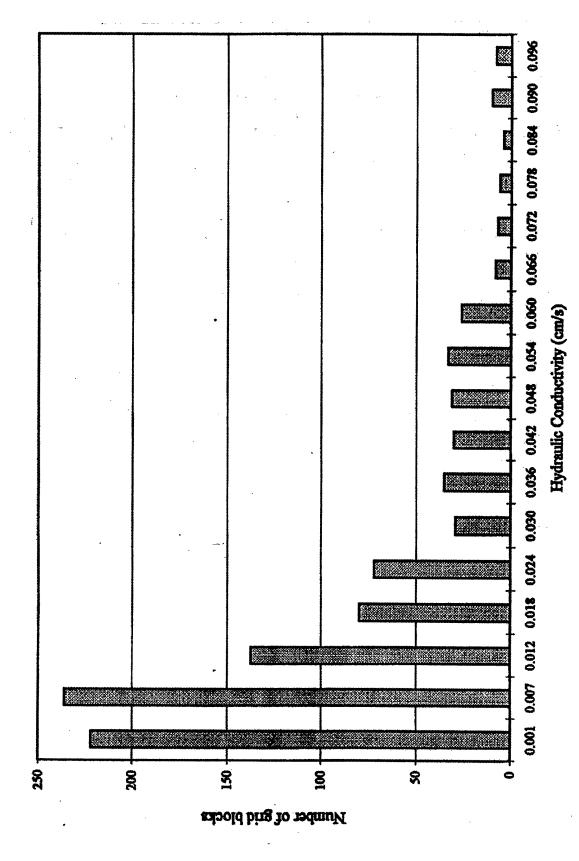


Figure 27. Estimated Hydraulic Conductivity Field in the Test Cell.



Histogram of the Estimated Hydraulic Conductivity Distribution. Figure 28.

was used as an initial guess. UTSTREAM then made adjustments to the saturation at each gridblock so that the simulated retardation factors of 2,2-dimethyl-3-pentanol were matched to the retardation factors from the field data.

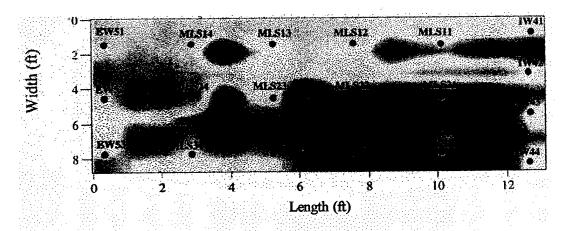
The final NAPL saturation distribution was obtained after a convergence of the objective function was achieved. Several plan and cross-sectional views of the resulting NAPL saturation distribution are shown in Figure 29 through Figure 32. Figure 33 shows the histogram of this NAPL saturation distribution. Based upon the NAPL saturation in each gridblock and gridblock size, it was estimated that a total volume of 394 liters (104 gallons) of NAPL was present.

The results from the method of inverse modeling are in good agreement with the results from the method of first temporal moment analysis. Both methods indicate that the average NAPL saturation in the test cell is approximately 5 percent. Although the total NAPL volume from the method of first moment analysis was approximately 469 liters (124 gallons), it was obtained by tracer data extrapolation up to 16 days. Therefore, it represents the NAPL volume in the saturated zone of the entire test cell. The estimated tracer swept volume was 9.3 m<sup>3</sup>. Because of the irregularly shaped boundary of the test cell, the simulation grid only represents the rectangular portion of the test cell between the rows of the injection and extraction well. The 394 liters (104 gallons) of NAPL from the inverse modeling, therefore, represent the NAPL volume in the pore space between the rows of the injection and extraction wells. Assuming a porosity of 0.28, this contains a pore volume of 8.19 m<sup>3</sup>.

The NAPL is non-uniformly distributed in the test cell ranging from 0 to 10 percent in saturation. The average NAPL saturation is higher in the intermediate layers of the test cell (5.3 percent for Red and 5.7 percent for White) compared to the top (4.2 percent black) and bottom (4.1 percent yellow) layers of the test cell. This kind of variation is also consistent with the results from the method of first moment analysis.

It should be noted, however, that although most of the tracer response data match the model predictions, the conductivity and NAPL saturation distributions obtained are not unique, and these results contain some uncertainties. There are many factors which may affect the final

# a) A plan view at the BLACK multilevel sampling points.



# b) A plan view at the BLUE multilevel sampling points.

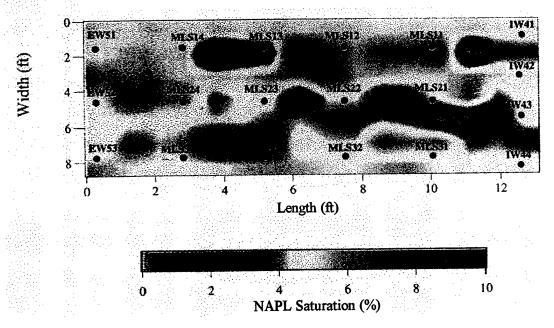
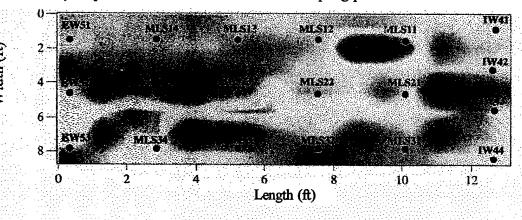


Figure 29. Plan view of Estimated NAPL Distribution in the Test Cell.

a) A plan vew at the RED multilevel sampling points.



b) A plan vew at the WHITE multilevel sampling points.

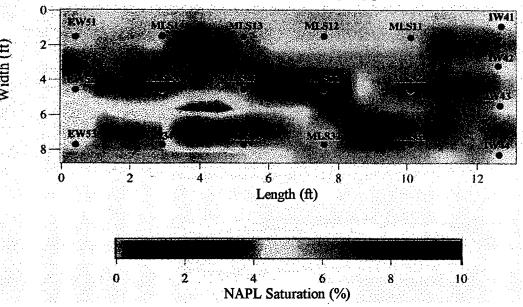
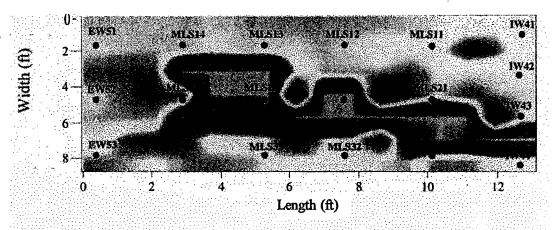


Figure 30. Plan View of Estimated NAPL Distribution in the Test Cell.

a) A plan view at the YELLOW multilevel sampling points.



b) A cross-sectional view along the extraction well EW51.

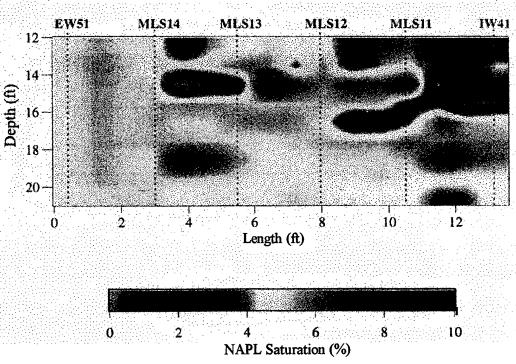
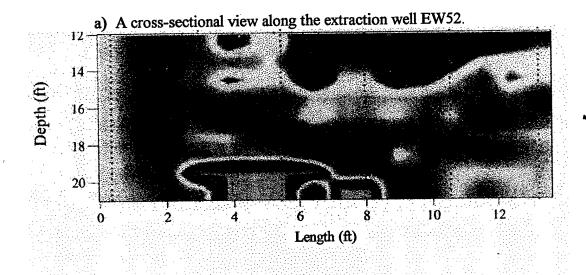


Figure 31. Estimated NAPL Distribution in the Test Cell.



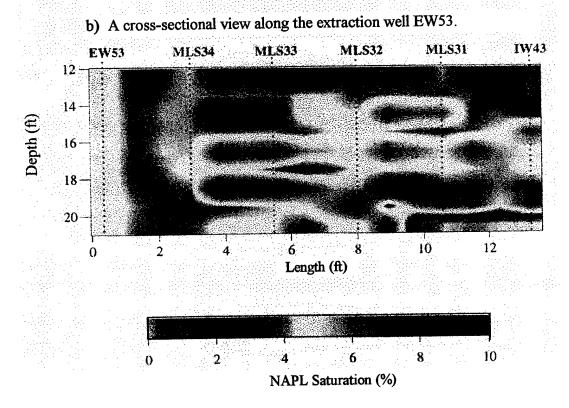
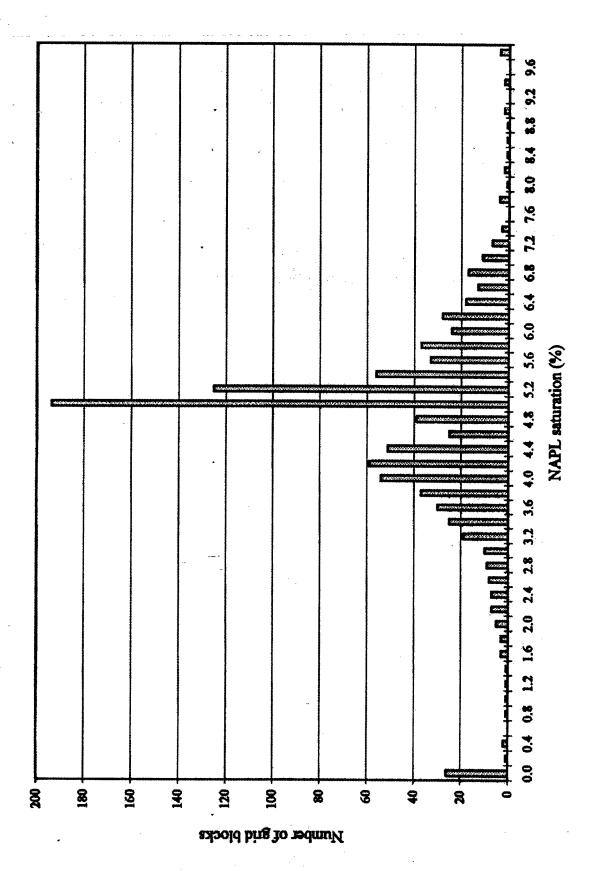


Figure 32. Estimated NAPL Distribution in the Test Cell.



Histogram Showing the Estimated NAPL Saturation Distribution, Figure 33.

results of inverse modeling. The geometry of the simulation grid, for example, contributes to the uncertainty of the accuracy of the results. The boundary of the test cell has an irregular shape and is not straight lines as in the model. Such an irregular shape would cause tracer particles to move slowly along the boundary. As a result, the tails of the tracer response curves from the field data for the extraction wells were prolonged compared to the model predictions.

Secondly, the CONJUGATE simulator uses the conjugate gradient method. The conjugate gradient method is an iterative method for finding the minimum of the objective function, and requires an initial guess close to the correct solution. The results presented in this report are the best results we obtained from a series of simulation runs with different initial guesses of hydraulic conductivity and NAPL saturation.

Thirdly, the quality of the tracer data also affects the accuracy of estimation. The tracer data used in this analysis contain tracer data from a total of 60 multilevel samplers and 3 extraction wells. The simulator treats the data from each point as having the same accuracy. Therefore, errors from one monitor point can have a big effect on the estimation results. Based on the experience of this data analysis, it is impossible to obtain a perfect match in each observation point.

Nonetheless, the estimated conductivity and NAPL saturation allow for a large part of the tracer data to be matched, therefore, they must be reasonably representative of the actual spatial distribution of hydraulic conductivity and NAPL saturation in the test cell. Although the results from inverse modeling contain some uncertainties, the combination of the results from the method of first temporal moment analysis and inverse modeling provide a reliable assessment of the hydraulic conductivity and NAPL saturation distributions.

#### 2. Posttreatment

ARA is currently awaiting the results of the posttreatment PITT data analysis, which is being performed by INTERA, Inc. These results will be forwarded upon receipt as an addendum.

# C. SOIL CORING RESULTS

#### 1. Stratigraphy

Based on visual descriptions of the soil corings collected during the pre- and posttreatment characterization activities, a detailed three-dimensional model of the soil stratigraphy was developed using the software package "EarthVision®." The model is represented in Figure 34 and illustrates a north-south profile and an east-west cross-section, as well as 3-D isometric view from a south-west perspective. The model shows clearly that there are four distinct stratigraphic sections within the cell consisting of three interbedded soil types; (1) poorly-graded sands, (2) well-graded gravely sand mix, and (3) clay. This appears to be consistent with previous investigations of the OU-1 site, which state that the aquifer is composed of interbedded silts, sands, and gravels of the Provo and Alpine Formations. Cross sections developed by Montgomery Watson indicate that an aquitard consisting of laminated clay with thin silt and sand interlaminae exists from depths of about 7.6 to 9.1 meters (25 to 30 feet) bgs. The top surface of the aquitard is very irregular and possibly represents an erosional surface on which the coarser-grained channel deposits of the Provo Formation were deposited.

# 2. Chemical Analyses

The soil samples collected during the pre- and posttreatment characterization were analyzed at Michigan Technological University (MTU) in accordance to RSKSOP-72 (Appendix A). The target analytes included:

TABLE 9. TARGET ANALYTES SELECTED FOR PRE- AND POST-SOIL CHARACTERIZATION.

1,1,1-Trichloroethane	1,3,5-Trimethylbenzene	
Trichloroethene	Decane	
Toluene	1,2-Dichlorobenzene	
m+p Xylene	Undecane, and	
o-xylene	Naphthalene	

MTU reported the results in units of mass of target analyte per unit volume of solvent (e.g., mg of target analyte/Liter of dichloromethane; mg/L). These values were converted to units of mass of target analyte per unit mass of dry soil (e.g., mg of target analyte/mass of dry soil; mg/kg) using the following relationship:

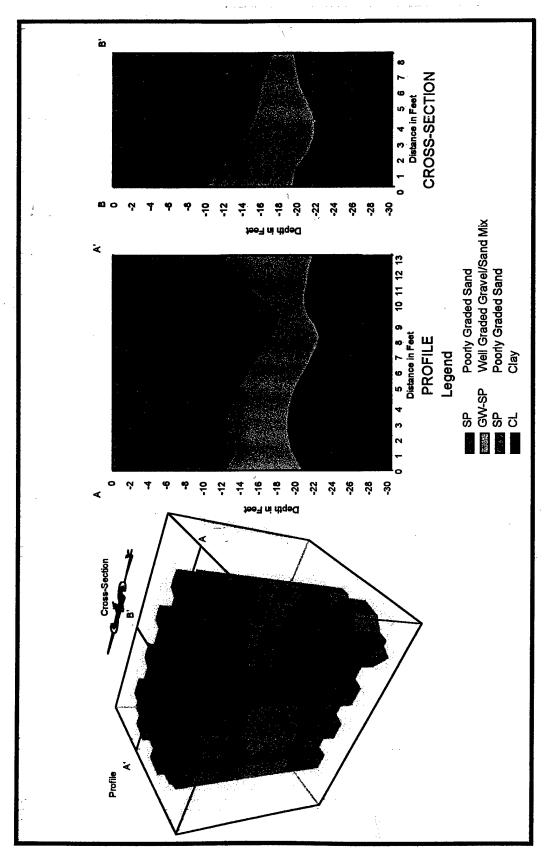


Figure 34. Relative Locations of Soil Borings and Resulting Stratigraphic Interpretations from Pre- and Posttreatment Soil Characterization Events.

The mass of the dry soil in the actual sample was back-calculated based on the moisture content of a duplicate sample. The soil moisture content data was determined at ARA's soil lab in accordance with ASTM Method D 4959. Appendix D presents the tabulated concentration results from both the pre- and posttreatment characterization efforts determined using the above calculations.

Once the data were processed, they were reviewed and two of the analytes considered to be representative of the target analyte list were modeled in three dimensions (3-D) using the software package "EarthVision®." Isometric views of the resulting 3-D interpretations of the decane and o-xylene distributions from the pre- and posttreatment soil characterization data are presented in Figures 35 through 38, respectively. Horizontal slices of these 3-D models, including the concentrations of all of the target analytes detected at the various locations and depths are presented in Appendix I.

The pre-treatment distribution of o-xylene throughout the cell at five discrete depths is illustrated in Figure I-2 in Appendix I. In general, the highest concentrations (e.g., 10-mg/kg to <20-mg/kg) appear to be concentrated in the 5.6- to 6.2-meter (18- to 20-foot) bgs region with isolated 'hot spots' located in the south-central and northwest portions of the cell. The highest concentration, measuring 31.4 mg/kg, was detected at location U1-2753 at a depth of 5.6 meters (18 feet) bgs. The profile and cross-sectional views of Figure 34 indicate that this stratigraphic section is the most permeable region of the cell, consisting of well-graded gravel/sand mix. This correlates with the estimated hydraulic conductivity field presented in Figure 27. Based on Figure 27, the hydraulic conductivity within this region is on the order of 0.05 to 0.08 cm/s.

# D. GROUNDWATER SAMPLING CHEMICAL ANALYTICAL RESULTS

There were four separate sampling events during this study to ascertain the chemical composition of the groundwater before and after steam injection treatment; two under dynamic flow

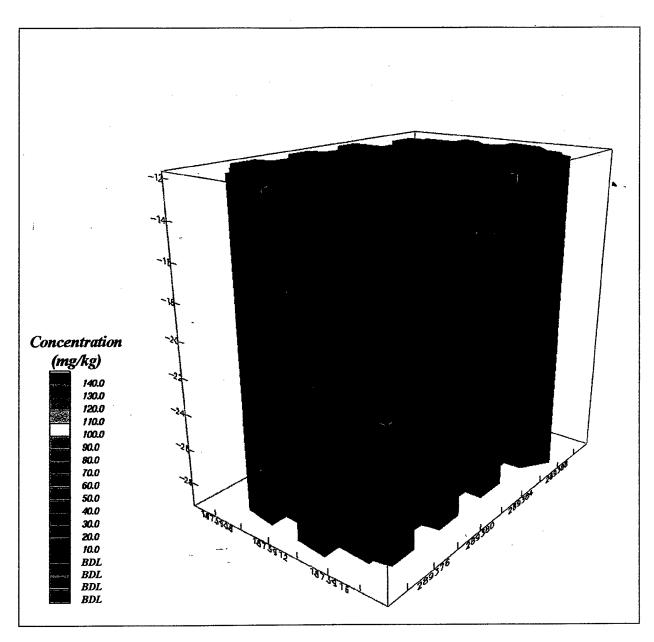


Figure 35. Three-Dimensional Interpretation of the Decane Distribution Within the Cell Based on the Results of the Pretreatment Soil Characterization Data.

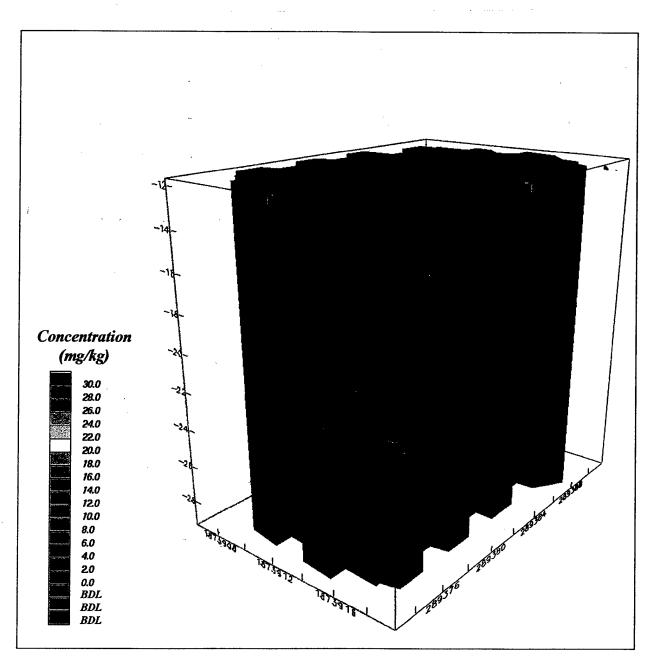


Figure 36. Three-Dimensional Interpretation of the o-Xylene Distribution Within the Cell Based on the Results of the Pretreatment Soil Characterization Data.

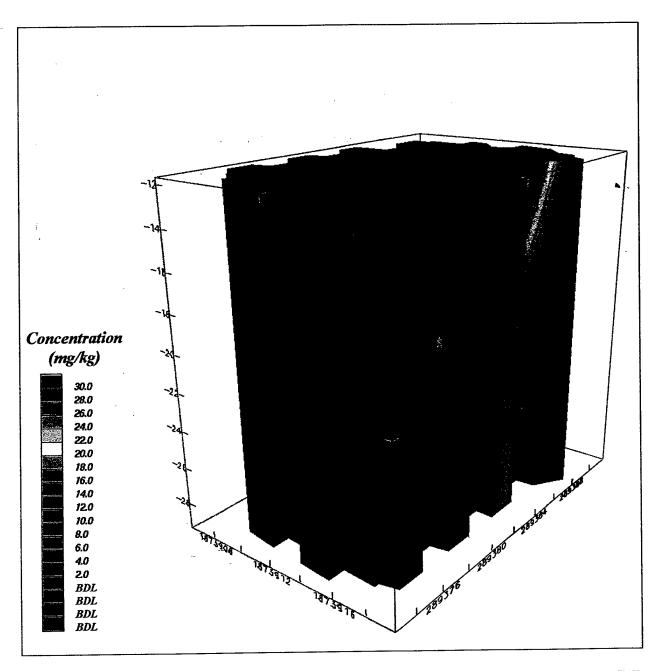


Figure 37. Three-Dimensional Interpretation of the Dacane Distribution Within the Cell Based on the Results of the Posttreatment Soil Characterization Data.

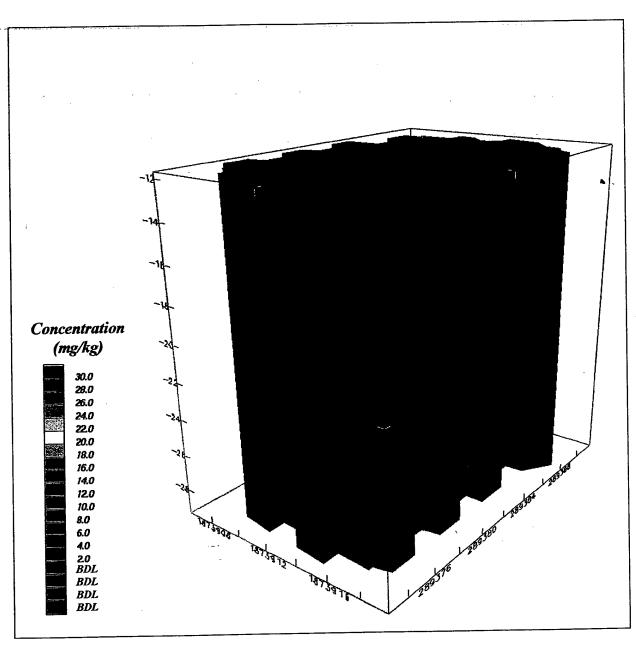


Figure 38. Three-Dimensional Interpretation of the o-Xylene Distribution Within the Cell Based on the Results of the Posttreatment Soil Characterization Data.

conditions (pre and posttreatment) and two under static conditions (pre and posttreatment). The following subsections are organized into two primary sections: Pre-, and Posttreatment Characterization. These sections are further divided into subsections that discuss the individual Dynamic and Static Sampling Events.

# 1. Pretreatment Groundwater Characterization

The purpose of the pretreatment sampling events was to develop a baseline of the groundwater quality from which to compare to the posttreatment results. The dynamic sampling event was conducted after achieving steady-state flow conditions just before beginning the preand post treatment PITTs. Static conditions are defined as the no-flow conditions within the cell after a minimum of 48 hours following the completion of the pre- and posttreatment PITTs.

#### a. Dynamic Sampling Event

Prior to beginning the pretreatment PITT, a steady-state flow regime was established within the test cell that was maintained throughout the duration of the PITT. After flushing approximately three pore-volumes of potable water through the cell, under steady-state flow conditions at an average rate of 4.1 liters per minute (lpm) (1.08 gallons per minute), a total of ten samples were collected from the MLS sampling grid and three from the extraction wells. The design of the MLS sampling system allowed for the collection of all of these samples simultaneously to provide a "snapshot" in time of the groundwater quality throughout the cell. The samples were stored in the mobile laboratory's refrigerator, where they were maintained at 4°C prior to shipment to RSKRL for analysis of the project target analyte list by Method RSKSOP-148. A copy of the Standard Operating Procedure (SOP) for Method RSKSOP-148 is included as Appendix F.

The results of the chemical analyses show that seven of the eleven target analytes were detected in at least some of the 13 locations sampled during this event. Vinyl chloride, 1,1,1-trichloroethane, trichloroethene, toluene, and 1,2-dichlorobenzene were the most prevalent compounds while m+p xylene and o-xylene were detected at only four locations each. In general, the concentrations of all of the analytes ranged from nondetectable (ND) to a high of 807-ppb 1,2-dichlorobenzene. The concentrations of vinyl chloride range from ND to 111-ppb at

Extraction Well U1-2751. This is noteworthy as it may be an indication that natural degradation of some of the other chlorinated compounds is occurring. The remaining target compounds, 1,3,5-trimethylbenzene, decane, undecane, and naphthalene were not found at any of the locations.

One clear observation from these results is that the highest concentrations of the target analytes occurs in the 6.1- to 6.7-meter (20- to 22-foot) zone. This depth correlates with the well-graded gravel sand mix strata as shown previously in Figure 34. It does not, however, correlate with the highest concentrations detected in the soils. The soils show higher concentrations in the 16- to 18-foot strata. One possible explanation for this discrepancy is that the NAPL is funneling through the most permeable strata resulting in higher concentrations of target analytes.

#### b. Static Sampling Event

Upon Completion of the PITT, the flow through the cell was terminated and water remaining in the cell allowed to equilibrate for 6 days. At that time, 37 samples from the MLS points, three from the extraction wells, and three from the injection wells were collected and delivered to RSKRL where they were analyzed in accordance with RSKSOP-148 for the target analyte list. As with the dynamic sampling event, the MLS samples were collected simultaneously using the MLS sampling system to provide a "snap-shot" of the groundwater quality at a discrete point in time. The results of these analyses are included in Appendix G.

The samples collected from the injection and extraction wells were collected using low-flow sampling techniques employing a peristaltic pump and a flow-through cell to monitor groundwater parameters for temperature, pH, conductivity, dissolved oxygen and turbidity. These parameters were recorded and the data are included in Appendix H.

Samples were also collected from the injection and extraction wells by Montgomery-Watson personnel for analysis of VOCs, BNAEs, Pesticides and PCBs, and dissolved Dioxins/Furans, and TPH. These data have not been released as of this date and will not be discussed further.

At least one or more of the target analytes were detected in each of the MLS sampling locations. This is also true for all of the injection/extraction wells with the exception of injection well U1-2743 where none of the target analytes were detected. Vinyl chloride, 1,1,1-trichloroethane, trichloroethene, and toluene were prevalent at most of the sampling locations. M+p Xylene and o-xylene were found at relatively few MLS sampling locations and were found exclusively at the 6- to 6.7-meter (20- to 22-foot) (bgs) sampling depths plus at the three extraction wells. This depth interval correlates with the interface between the well-graded gravel/sand mix and the poorly-graded sand stratigraphic sections.

The analytes 1,3,5-Trimethylbenzene, decane, undecane, and naphthalene were all undetected at all of the MLS sampling locations but were detected in the three extraction wells. This may be an indication that pools of NAPL are located within the cells such that the MLSs are not detecting them.

In general, inspection of the results show that the levels of the target analytes were higher during the static sampling event as compared to the dynamic event, particularly for the heavier compounds such as decane, 1,2-dichlorobenzene, undecane and naphthalene. This trend is illustrated in Figures 39 through 41, which show the comparison of the results from the pretreatment static and dynamic sampling events at the three extraction wells (e.g., U1-2751 through U1-2753). Intuitively, this makes sense, since for the static sampling event, the target analytes have been allowed significantly more time to partition into the groundwater prior to sampling.

#### 2. Posttreatment Groundwater Characterization

After the steam injection/soil venting treatment was completed, the cell was allowed to cool to approximately 22°C. The cooling process within the cell was accelerated by establishing the flow of potable water required to conduct the posttreatment PITT. This promoted the removal of residual heat from within the cell, and provided an opportunity to establish a dynamic equilibrium inside the cell.

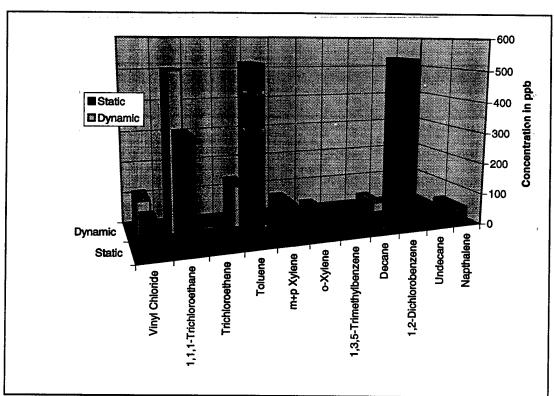


Figure 39. Comparison of Groundwater Sampling Results from the Pretreatment Static and Dynamic Sampling Events at U1-2751 Extraction.

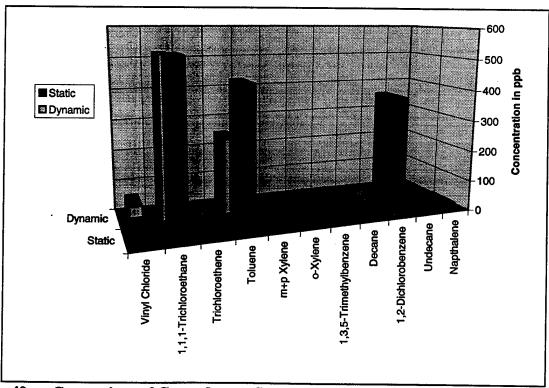


Figure 40. Comparison of Groundwater Sampling Results from the Pretreatment Static and Dynamic Sampling Events at U1-2752 Extraction.

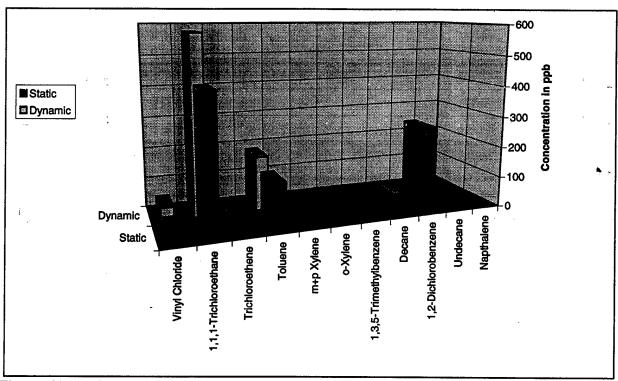


Figure 41. Comparison of Groundwater Sampling Results from the Pretreatment Static and Dynamic Sampling Events at U1-2753 Extraction.

#### a. Dynamic Sampling Event

Approximately 2.5-pore volumes of potable water were flushed through the cell at a rate of 3.6 liters per minute (lpm) (.95 gallons per minute) prior to collecting the posttreatment dynamic groundwater samples. A total of 52 samples, three from the extraction wells and 49 from the MLS sampling grid, were collected for chemical analysis of the target analytes. As with the pretreatment sampling events, the MLS samples were collected simultaneously to provide a "snapshot" in time of the groundwater quality throughout the cell. These samples were immediately stored in coolers, then moved to the mobile laboratory where they were stored in a refrigerator and maintained at 4°C prior to shipping to RSKRL for analysis using EPA Method RSKSOP-148. The results are included in Appendix G and are discussed in the following paragraphs.

Chemical analysis of the groundwater samples collected during this sampling event show a significant reduction in the levels of the target analytes relative to pretreatment dynamic sampling event. This is clearly illustrated in Figures 42 through 44, showing a comparison of the

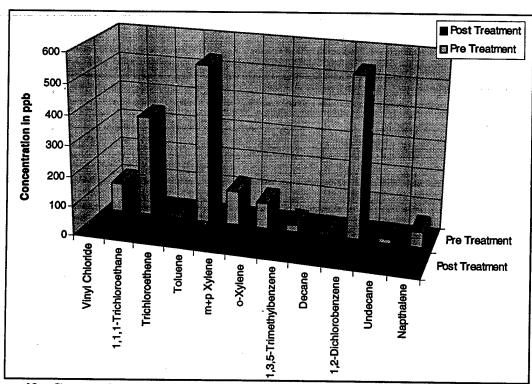


Figure 42. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Dynamic Sampling Events at U1-2751.

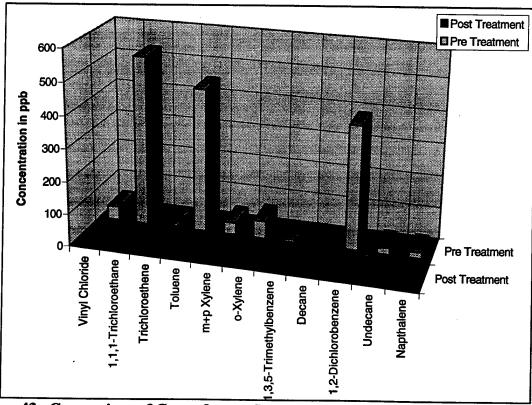


Figure 43. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Dynamic Sampling Events at U1-2752.

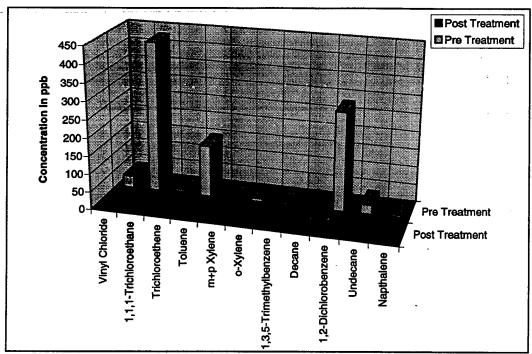


Figure 44. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Dynamic Sampling Events at U1-2753.

pre- and posttreatment dynamic sampling from each of the extraction wells. These data show that vinyl chloride and undecane have essentially been removed from the cell; vinyl chloride was detected only once at location U1-2734 at the 4.3-meter (14-foot) depth, while undecane was not detected in any of the sampling locations. M+p xylene, 1,3,5-trimethylbenzene, decane and naphthalene were detected at only three or four locations and at relatively low concentrations. Trichloroethene appears to be the most prevalent target analyte detected during this event with values ranging from non-detect to 14.9-ppb. 1,1,1-trichloroethane, toluene, o-xylene, and 1,2-dichlorobenzene were consistently found together at approximately ten MLS sampling locations. Relatively high levels of 1,2-dichlorobenzene were detected in four MLS sampling locations with values ranging from 834-ppb to 324-ppb.

One noteworthy observation of these data, besides the fact that they are significantly reduced relative to the pretreatment sampling events, is that the target analytes detected at the MLS stations do not appear to be reaching the extraction wells. This may be indicative of soil regions bypassed by the flowing steam from which the NAPL was not effectively removed.

### b. Static Sampling Event

The final groundwater sampling event occurred after completion of the posttreatment PITT. Once the flow through the cell was shut down, the water within the cell was allowed to equilibrate with the surrounding aquifer for a period of approximately 66 hours. At that time, a complete round of groundwater samples was collected to provide a final 'snapshot' of the water quality within the cell. A total of 63 samples were collected from the MLS sampling grid and the six injection/extraction wells. These samples were handled and analyzed in the same manner as in the previous sampling events. The results of the analyses are included in Appendix G and will be discussed below.

A close look at the results (Figures 45 through 47) shows that more volatile compounds such as vinyl chloride, 1,1,1-trichloroethane, trichloroethene and toluene were significantly reduced in the posttreatment groundwater samples. This reduction is particularly apparent in the concentration of 1,1,1-trichloroethane, where pretreatment concentrations ranged from 500-600 ppb and posttreatment concentrations were reduced to less than 100 ppb. Some of the less volatile compounds such as the xylenes, 1,3,5-trimethylbenzene and 1,2-dichlorobenzene show increased concentrations in the posttreatment groundwater samples, particularly with regards to the 1,2-dichlorobenzene. This increase is most likely due to the increased mole fraction of these compounds in the NAPL by the steam leaving higher mole fractions of the less volatile compounds and therefore higher equilibrium groundwater concentrations. Interestingly, since these compounds are sparingly soluble in water only a small amount of NAPL remaining in the cell is required to yield the high observed groundwater concentrations; hence these groundwater concentrations are not indicative of the NAPL saturation remaining in the cell.

The injection and extraction wells were sampled using peristaltic pumps as described in § IV.D.1.b. Duplicates of these samples were collected by Montgomery-Watson and analyzed for VOCs, BNAEs, Pesticides and PCBs, dissolved Dioxins/Furans, and TPH. As discussed previously, these results are not available at this time and will not be discussed further.

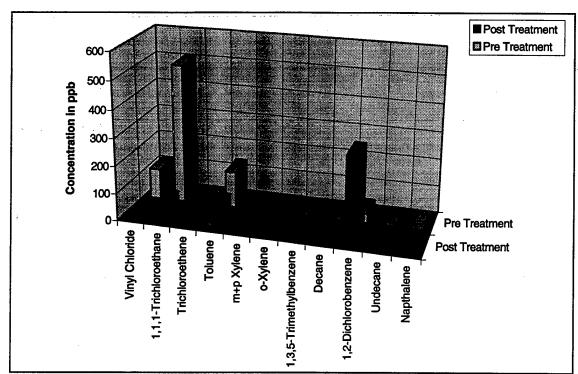


Figure 45. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Static Sampling Events at U1-2751.

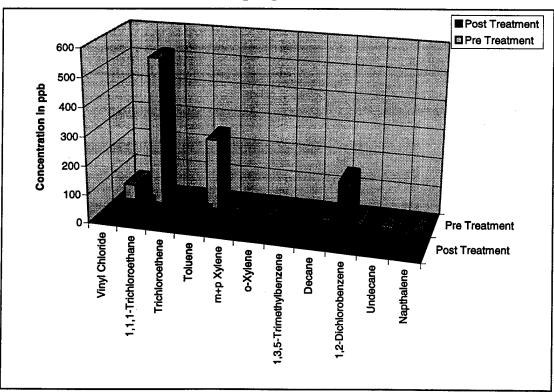


Figure 46. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Static Sampling Events at U1-2752.

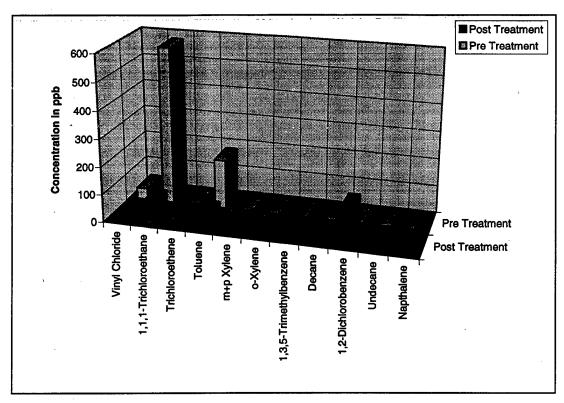


Figure 47. Comparison of Groundwater Sampling Results From the Pre- and Posttreatment Static Sampling Events at U1-2753.

#### E. SOIL VAPOR EXTRACTION

#### 1. Cell Dewatering

As discussed in Section III, the test cell was dewatered before initiating soil vapor extraction. A total of 1,770 liters (468 gallons) of groundwater were extracted, lowering the water table 1.34 meters (4.4 feet) to a depth of about 6.7 meters (22 feet) below ground surface. The leakage rate of surrounding groundwater into the cell was also evaluated during this period. In 2 days of monitoring, the water level did not change significantly, indicating cell leakage was very low. Leakage had been observed previously, but during a season when the water table was about 4 feet higher.

### 2. Soil Vapor Extraction Results

Soil vapor extraction was initiated in the test cell after dewatering to provide baseline concentrations for evaluating the enhancement in contaminant recovery by steam injection. The SVE testing lasted 73 hours and included four periods of extraction separated by three periods of

no flow to allow vapor concentrations to rebound. The periods are described in Table 5. The total air extraction rate from the test cell during the SVE testing is plotted in Figure 48. The measured total extraction rate varied between 0.60 standard cubic meters per minute (scmm) (21 cubic feet per minute) and 0.71 scmm (25 cubic feet) when the system was operating. The vacuums applied to the wellheads to achieve these flows were 483 millimeters of water column (mmH<sub>2</sub>O) (19 inches of water column [inH<sub>2</sub>O]) during the first period, 432 mmH<sub>2</sub>O (17 inH<sub>2</sub>O) during the second, 559 mmH<sub>2</sub>O (22 inH<sub>2</sub>O) during the third, and 559 mmH<sub>2</sub>O (22 inH<sub>2</sub>O) during the fourth. As noted in Table 5, the center injection well was open during the first and second tests and closed during the third and fourth tests. The wellhead vacuums were smaller and the extraction flow rate was higher when the center injection well was open. The total extraction flow rate was about 8 percent lower when the injection well was closed.

During open injection well tests, the air flow drawn into the cell through the center well was about 0.19 scmm (6.7 scfm) or almost 25 percent of the total. The other 75 percent was drawn from the surface down. Vacuums in the cell during SVE were measured at the MLSs. The measured vacuums were used with a three-dimensional subsurface air flow model to estimate the air permeabilities of the test cell during SVE. The values were estimated to be 0.0193 cm/s and 0.00676 cm/s for the horizontal and vertical permeabilities, respectively. These permeabilities are typical of sands and gravels and the horizontal value matches extremely well with the arithmetic average of 0.027 cm/s estimated by the inverse modeling technique. The permeability of the de-watered soil to air is expected to be less than to water since the soil remains moist. The ratio of the measured horizontal permeabilities yields an air relative permeability of 0.72, which indicates an approximate residual water saturation of 20 percent after de-watering.

Vapor concentrations of 12 of the target compounds measured with the on-site GC during SVE are plotted versus time in Figure 49. The concentrations of the more volatile compounds such as 1,1,1-trichloroethane (1,1,1-TCA) and heptane were initially high and exhibited the exponential decay characteristic of long-term SVE. The concentrations of these compounds dropped about 80 percent during this short period of testing. For moderately volatile compounds such as toluene and nonane, the vapor concentrations appeared to decrease slightly during the tests. Concentrations of compounds with relatively low volatility, such as 1,2-dichlorobenzene (1,2-DCB) and undecane, were erratic and did not appear to decrease during the SVE testing.

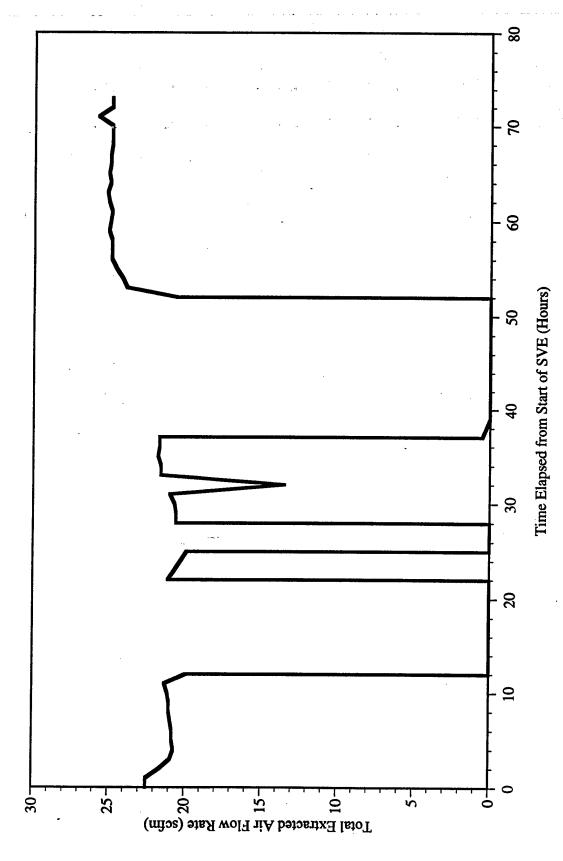


Figure 48. Total Extracted Air Flow Rate During SVE.

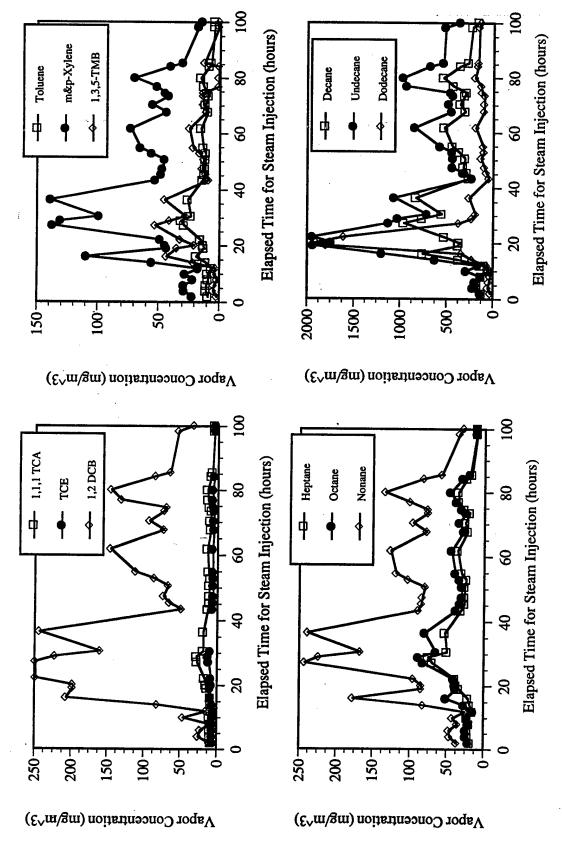


Figure 49. Vapor Concentrations During Steam Injection.

Measured concentrations of the compounds were variable, and this is attributed to the changing content and distribution of moisture in the cell, since QA/QC sampling verified the repeatability of the sampling procedure.

For an extraction rate of 0.62 scmm (22 scfm) and an effective porosity of 35 percent, the test cell pore volume was flushed about every 45 minutes. As observed in Figure 49, this period corresponds to the extraction time required to reach the peak vapor concentration after startup for each extraction period. Moderate concentration rebounds are evident at the start of test periods 2 and 4 after brief periods of shutdown. Given the short shutdowns, the rebound was probably the result of vapor diffusion from NAPL-contaminated zones not in good contact with the flowing air rather than a liquid-liquid diffusion limitation, which would require much longer equilibration periods.

The mass of target compounds removed during the SVE test was estimated from the measured extraction rate and the measured concentrations. These calculated masses are presented below in Table 10. During the pre-steam SVE, 2,030 cubic meters (71,672 cubic feet) of air was extracted from the test cell. This volume yields an average total target compound concentration of 445 mg/m<sup>3</sup>. The total period of extraction in this phase was 47 hours, yielding an average target mass removal rate of 19 grams (0.7 ounce) per hour.

#### F. STEAM-ENHANCED EXTRACTION

After the dewatering and soil vapor extraction tests, steam injection was initiated to assess the increase in contaminant removal provided by heating. Steam was injected in the Center Well U2-2771, while dual-phase extraction was applied in the four corner wells. After several hours of injection, the steam began reaching the extraction wells. The time for steam to reach different extraction wells varied because of heterogeneities in the cell soil. After steam had reached all four extraction wells, steady steam injection was continued. The discussions of the steam injection and contaminant recovery are presented in two parts: the initial soil heating, and the subsequent quasi-steady steam injection and extraction.

TABLE 10. CUMULATIVE MASS OF TARGET COMPOUNDS REMOVED DURING SVE.

Target Compound	Pre-Steam SVE Cumulative  Mass Removed		
	(grams)	(pounds)	
Hexane	16.1	0.04	
cis-1,2-DCE	67.7	0.15	
1,1,1-TCA	32.8	0.07	
Heptane	78.3	0.17	
Benzene	0.3	0.00	
TCE	11.9	0.03	
Octane	48.2	0.11	
Toluene	22.6	0.05	
Nonane	67.3	0.15	
m+p-Xylene	45.4	0.10	
o-xylene	12.6	0.03	
Decane	173.4	0.38	
1,3,5-TMB	4.2	0.01	
Undecane	254.4	0.56	
1,2-DCB	17.1	0.04	
Dodecane	51.0	0.11	
		·	
Total	903.5	1.99	

### 1. Initial Cell Heating

Steam injection followed an extended period of SVE with the center well closed. Just before the start of injection, the center well was under a vacuum of 490 mm $H_2O$  (19.3 in $H_2O$ ), which decreased to 241 mm $H_2O$  (9.5 in $H_2O$ ) after steam injection started. The steam injection rate was roughly steady at 113 kg/hr (250 pounds per hour) throughout the steam injection phase and the well remained under a vacuum. The steam injection well was screened from 3.8 to 7.6 meters (12.5 to 25 feet) below ground surface (bgs). The water level in the cell was at a depth of

about 6.75 meters (22 feet) when steam injection was initiated. The initial growth of the steam zone was tracked with thermocouples installed with the MLSs. Based on the thermocouple measurements, the steam preferentially flowed toward the south end of the cell. The steam zone appeared uniformly in MLS U1-2722 spaced 0.4 meters (1.3 feet) from the injection well in less than 30 minutes of injection. In contrast, at MLS U1-2723, spaced 0.4 meters to the north of the injection well, the steam reached the MLS after 3.6 hours of injection. As illustrated in Figure 50, steam flow to the north was also not vertically uniform. Steam first reached MLS U1-2723 at a depth of about 4.4 meters (14.4 feet). Steam then reached this MLS at depths of 3.54 meters (11.6 feet) and 5.84 meters (19 feet) after about 5 hours of injection. The soil interval at about 5.33 meters (17.5 feet) did not reach steam temperature until long after steam injection was initiated. The geologic logs in the vicinity of this MLS identify a poorly graded sand layer at this approximate depth surrounded by well-graded gravels. The sand interval has a lower permeability and provides a greater resistance to steam flow forcing the steam to move through the gravel layers, as observed in Figure 50. As discussed later, the contaminant concentrations were also highest nearest this sand interval. This indicates the NAPL was concentrated atop or within this sand. A high, immobile NAPL concentration further reduces the permeability of this material and inhibits steam flow through it. Temperature profiles from MLSs 21 and 33 also illustrate the impact of heterogeneities on the steam zone growth (see Figures 51 and 52). The steam bypasses lower permeability lenses or zones with high, immobile NAPL saturations, which must be heated by conduction. In addition, heating the residual NAPL results in distillation of volatile components, which acts as a heat sink, further inhibiting a temperature increase in a NAPL-contaminated zone.

The times for steam to reach other locations across the test cell are summarized in Table 11 and shown graphically in Figure 53. This figure illustrates the preferential flow toward the southeast extraction well (U2-2741). Steam reached Extraction Well 41 after approximately 11 hours of injection and Well U1-2744 after about 14 hours. The steam zone reached Extraction Wells U1-2751 and U1-2753 after about 20 and 21 hours, respectively. This preferential southerly flow is in direct contrast to the results of the inverse modeling that estimated the soil permeability to be higher to the north around well U1-2751 as shown in Figure 27. This discrepancy could be explained by anisotropic permeability in the horizontal plane, since the

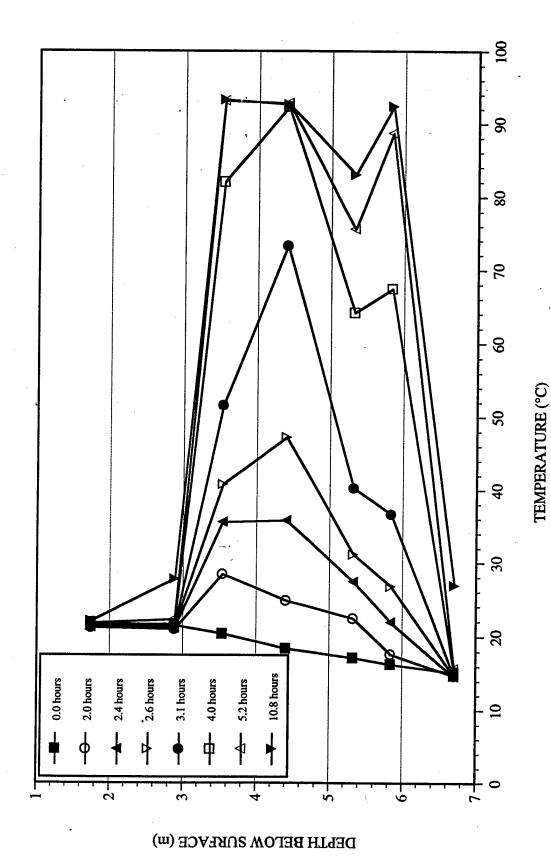


Figure 50. Temperature Profiles in MLS 23 During Initial Steam Injection.

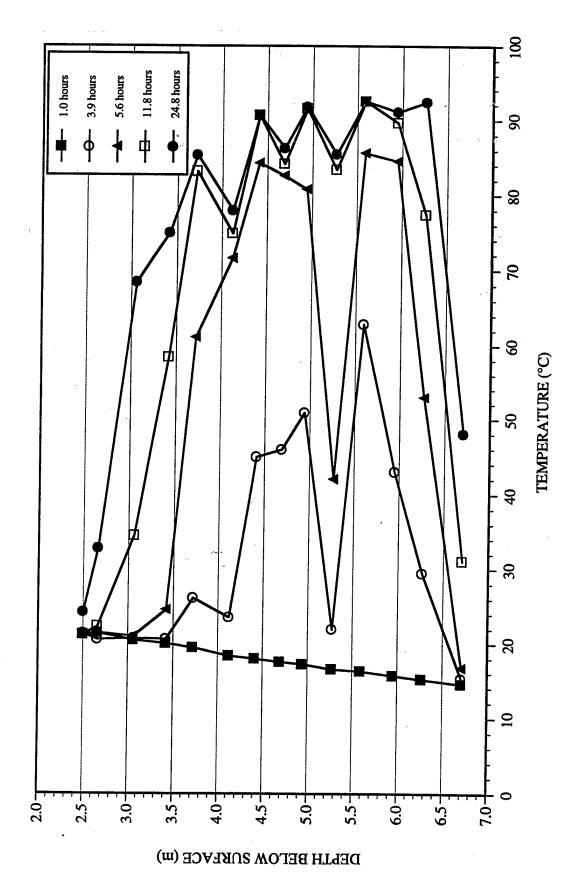


Figure 51. Temperature Profiles in MLS 21 During Initial Steam Injection.

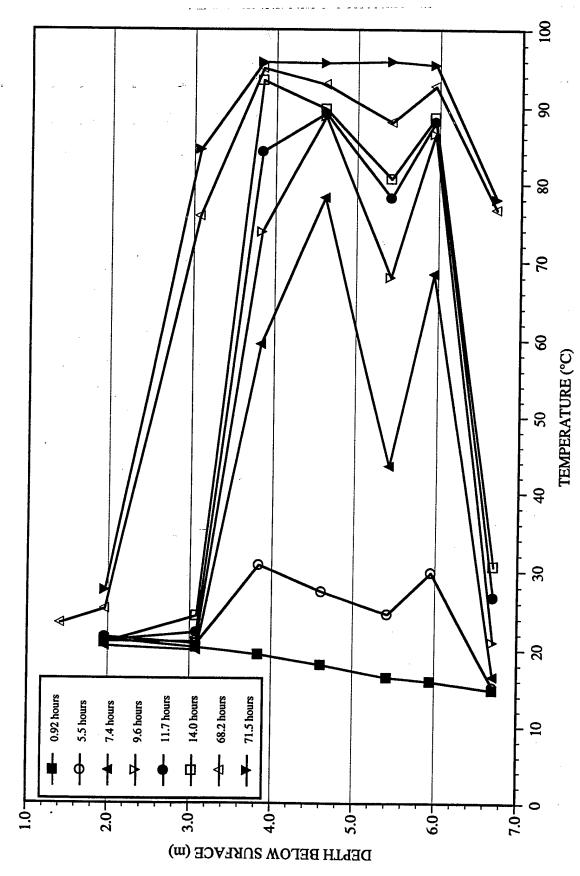


Figure 52. Temperature Profiles in MLS 33 During Initial Steam Injection.

TABLE 11. TIME FOR STEAM TO REACH VARIOUS CELL LOCATIONS.

Location	Distance from Injection Well (meters)	Time for Steam to Reach Location (hours)
" 11	1.43	7.5
12	0.86	1.6
13	0.86	3.9
14	1.43	14.0
21	1.21	5.6
22	0.39	< 0.6
23	0.39	3.6
24	1.21	11.7
31	1.43	8.8
32	0.86	6.7
33	0.86	9.6
34	1.43	15.8
41	2.24	11
44	2.24	14
51	2.13	20
53	2.13	21

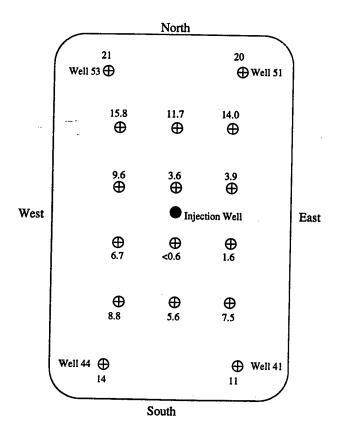


Figure 53. Illustration of Time in Hours for Steam to Reach Various Cell Locations.

flow directions differed in the two tests, but no data exist to support this conclusion. Shortly after steam breakthrough in all four extraction wells, the flow became effectively steady with the steam extracted nearly equaling the steam injected. The steam breakthrough times for the extraction wells were determined from measurements of the vapor temperature at the wellhead. The temperature histories for the extraction wells are plotted in Figure 54. Steam breakthrough was defined as the time when the extracted vapor temperature reached 50°C, since heat losses occur in the well casing and the temperature rise is sharp. The vapor temperature does not suddenly rise to steam temperature, because the steam initially condenses in the well and mixes with ambient air entering the well. After the well casing reaches steam temperature, the vapor temperature still continues to rise slowly as an equilibrium proportion of extracted steam and ambient air is approached. The vacuum at the extraction wellhead also changes during steam injection as shown in Figure 55. The vacuum histories illustrate the rise in vacuum that occurs when steam enters the well. The steam reduces the screen area available for ambient air to enter the well, and steam initially condenses in the casing, both of which tend to increase the vacuum in the well. Figure 55 also shows the vacuum increasing in Wells 51 and 53 before steam reaches these wells. This increase is caused by the reduced air flow in Wells 41 and 44, which raises the overall vacuum produced by the blower.

The total extracted flow rate of non-condensable gas (air) was also measured during this phase downstream of the condenser. The air flow from individual wells could not be measured because of the mixing with steam. The total extraction flow rate is presented in Figure 56. The total air flow rate remained relatively steady through steam breakthrough in Wells 41 and 44, but rose sharply just before breakthrough in Wells 51 and 53 at 20 hours of steam injection. This increase in total flow corresponds to the rise in vacuum produced by the reduced area for air flow, as discussed above.

Based on laboratory studies and field results from enhanced oil recovery, a bank of NAPL preceding steam breakthrough was possible. Yet, only about 2.5 gallons of NAPL were pumped off the top of the NAPL/water separator after steam breakthrough. As discussed earlier, the bank may have been larger, but the separator could have been inefficient because of an emulsion created by the water circulation pump. In any case, the steam injection was not effective at

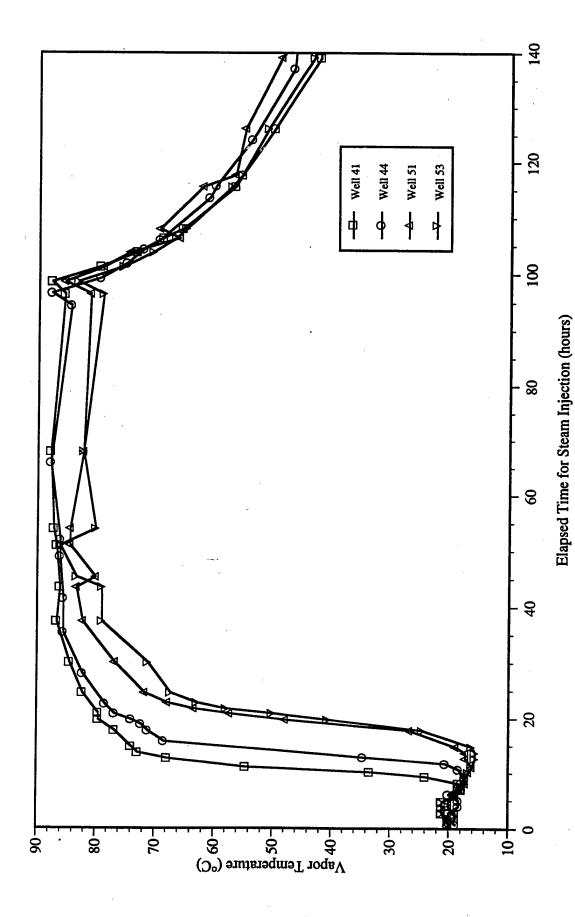


Figure 54. Extraction Well Temperature Histories during Steam Injection.

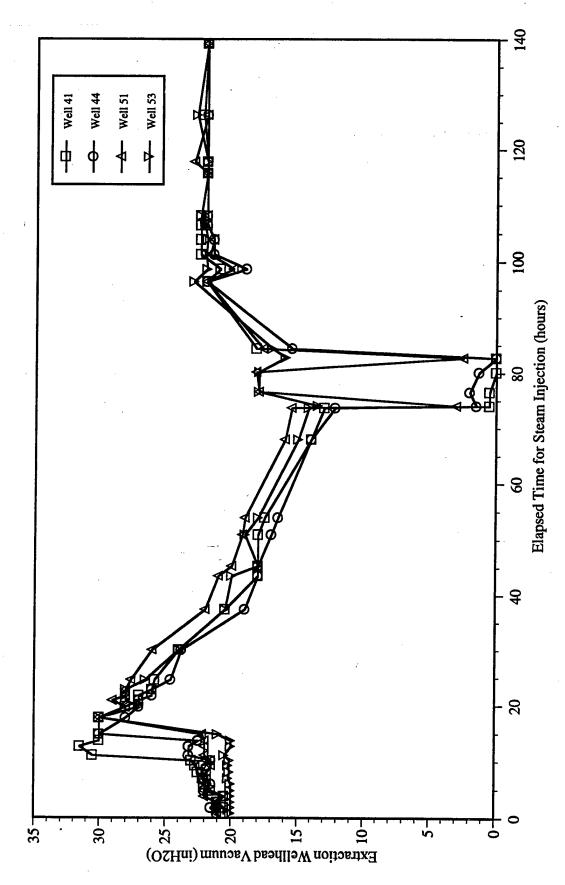


Figure 55. Extraction Well Vacuum Histories during Steam Injection.

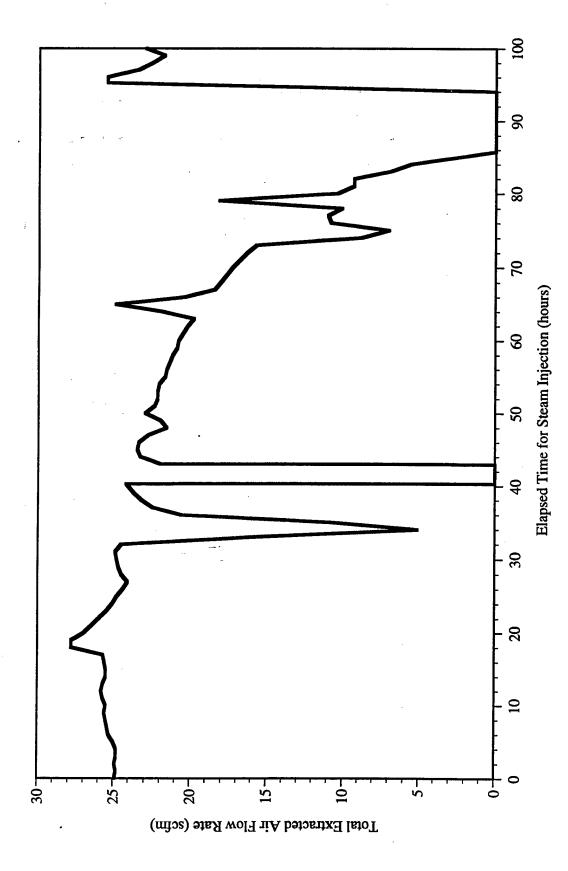


Figure 56. Total Extracted Air Flow Rate during Steam Injection.

driving the residual NAPL out of the cell. This result probably occurred because the viscosity of the NAPL was too high and the saturation too low to allow the formation of a stable NAPL bank ahead of the steam condensation front. Theory predicting the maximum NAPL viscosity that allows a stable NAPL bank to mobilize is developed in Appendix I. The theory reveals a surprising result that the steam injection rate does not influence the maximum NAPL viscosity for a stable drive. This result occurs because as the steam injection rate increases, the velocity of the NAPL bank increases proportionally. Therefore, the ratio of the vapor to NAPL pressure gradient does not change. This leads to the conclusion that increasing the injection rate does not extend the applicability of a steam drive to more viscous NAPLs. Using typical properties for Operable Unit One, the maximum NAPL viscosity allowing stable displacement by steam injection is about 2.5 centipoise (cP) at an injection quality of about 0.43. The NAPL at Operable Unit One has a viscosity significantly higher than 2.5 cP and therefore is not expected to be displaced. In addition, the steam injection during the field study was maintained at a quality close to one that has a maximum NAPL viscosity of only one cP for stable displacement. Therefore, the primary recovery mechanism is expected to be distillation of the NAPL components, which is discussed later.

#### 2. Quasi-Steady Steam Injection and Extraction

After steam breakthrough in all four extraction wells, the injection of steam into the cell and the discharge of water from the process equipment were nearly balanced. Heating in the cell was limited to bypassed, low-permeability regions. The downhole pumps for liquids were turned off, because very little, if any, fluids were being pumped from the wells. A mass balance for water in the test cell is illustrated in Figure 57. The time scale is set to zero at the start of SVE. Data at negative times corresponds to the initial groundwater pumping to dewater the cell, and illustrates the initial cell dewatering of 1,770 liters (468 gallons). No water was extracted during the SVE testing. As shown by the steam injected data, steam injection began at 73 hours and ended at 173 hours. The steam injection rate was essentially steady, but before steam breakthrough, the liquid discharge rate was less. This resulted in a net increase in water in the cell as illustrated by the dip in the water removed from the cell around 89 hours (i.e., after 16 hours of steam injection). At this time, a bank of steam condensate from heating the soil arrived at the extraction wells and was pumped out. After steam breakthrough in all four extraction

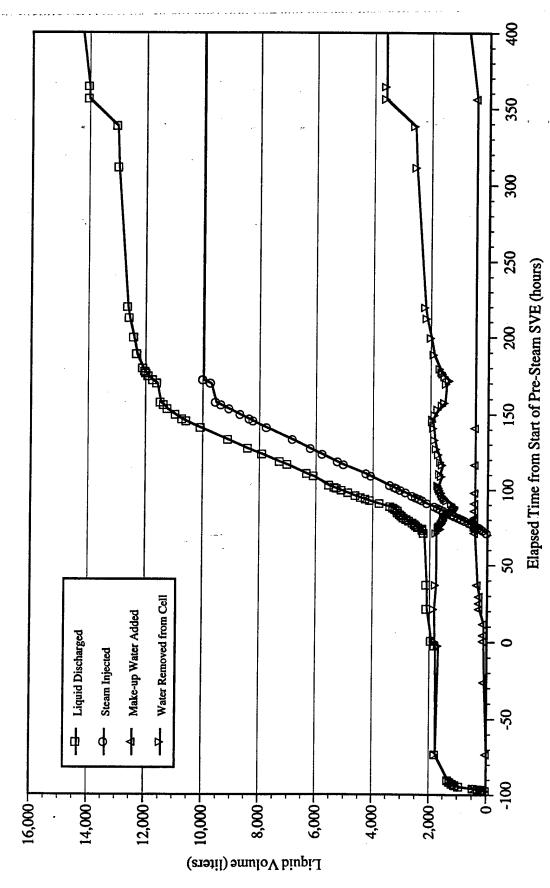


Figure 57. Mass Balance for Extraction and Injection of Water.

wells, the discharge rate slightly exceeded the steam injection rate and the cell was dewatered by an additional 150 liters (40 gallons) over the initial groundwater pumping. The additional dewatering probably resulted from evaporating water out of low permeability regions. A second dip in the water removed from the cell occurred around 146 hours (i.e., after 73 hours of steam injection) when a test of variable extraction rates in individual extraction wells was undertaken,

which is described later in this section. In summary, at the end of steam injection a total of 11,735 liters (3,200 gallons) of water had been discharged from the process equipment, 9,971 liters (2,634 gallons) had been injected as steam, and 437 liters (115 gallons) had been added to the process stream as makeup water leaving a net volume removed from the test cell of 1,327 liters (351 gallons).

The total extracted air flow rate from all four extraction wells during steam injection is given in Figure 56 and shows a gradual decrease in extracted air after steam breakthrough. The plot also shows three periods when electrical outages on the base shut the system down (i.e., 33 hours, 41 hours, and 90 hours). Variations in the total extraction rate are observed from 73 to 83 hours when variable flow testing occurred. The general trend was of a decreasing total air flow. The decreasing rate resulted from a combination of condensate collecting in extraction lines and steam vapor gradually displacing air flow into the extraction wells. During the electrical shutdown at 85 hours, the extraction lines were drained and the flow rebounded to near initial rates, but began dropping until steam injection was terminated at 100 hours. If operated longer, the system would be expected to reached an equilibrium point for the proportion of air extracted compared to the steam extraction rate.

Vapor concentrations of 12 of the target compounds measured with the on-site GC during steam injection are plotted versus time in Figure 58. The concentrations during the first 10 hours of steam injection, before steam breakthrough, showed no significant change from the SVE concentrations. Even after steam breakthrough, the most volatile of the target compounds (e.g., 1,1,1-TCA; heptane; TCE) did not increase significantly in concentration with the temperature increase. This indicates these compounds are probably not dissolved in the NAPL or exist primarily in the vadose zone above the original water table. To further illustrate this point, the maximum vapor concentrations of the target compounds detected during SVE and steam

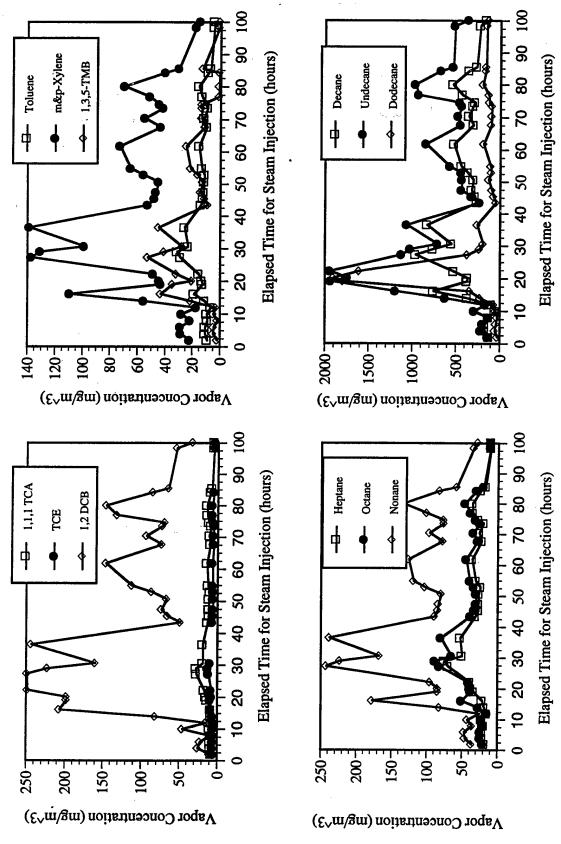


Figure 58. Vapor Concentrations During Steam Injection.

injection are compared in Table 12. The compounds are listed from most volatile (hexane) to least volatile (dodecane). The peak concentration of the volatile compounds during early SVE was higher than the maximum concentration detected during steam injection. The maximum concentrations during SVE are expected to be close to equilibrium values for the soil, groundwater, and contaminant mixture, since the cell had undergone very little extraction before SVE began. During SVE, the volatile contaminants were stripped from the exposed, residual groundwater, since the cell had been dewatered. The comparison of maximum concentrations also indicates disequilibrium within the NAPL (i.e., a non-uniform mixture), which would result from weathering of the NAPL over time. The more volatile compounds are more readily transported away. In addition, the increased temperature is not expected to increase the concentration of volatile compounds as much as semivolatile compounds, as indicated by the last column in Table 12 where the relative increase in vapor pressure is listed. The concentrations of moderately volatile compounds such as octane, toluene, nonane, and xylenes showed a significant increase with the increase in temperature, but not nearly as much as would be predicted by the increase in their vapor pressures. The weathering and disequilibrium are expected to be at a lesser degree than the volatile compounds, as was observed in the data. For the semi-volatile compounds such as 1,2-DCB, undecane, and dodecane, the vapor concentrations were much increased during steam breakthrough, although the concentrations dropped to apparent steady state values after steam breakthrough. These steady values remained much higher than the ambient SVE concentrations but still remained much less than would be predicted from vapor pressure increases.

The mass of target compounds removed during the steam injection was calculated from the measured extraction rate and the measured concentrations. These calculated masses are presented below in Table 13. During the steam injection, 3,194 cubic meters (112,788 cubic feet) of air was extracted from the test cell. This volume yields an equivalent average total target compound concentration of 1900 mg/m³ in the air (neglecting the steam vapor). The total period of steam injection and extraction was 100 hours yielding an average target mass removal rate of 60.7 grams (2.14 ounces) per hour.

TABLE 12. COMPARISON OF MAXIMUM VAPOR CONCENTRATIONS DURING SVE AND STEAM INJECTION.

Target Compound	Maximum Vapor Concentration (mg/m <sup>3</sup> )		Ratio of Steam to SVE Concentration	Ratio of Steam to SVE Vapor Pressure
	SVE	Steam		
Hexane	40.8	17.5	0.43	
cis-1,2-DCE	143.9	47.3	0.33	14
1,1,1-TCA	53.4	27.3	0.51	16
Heptane	101.6	74.8	0.74	22
Benzene	4.0	2.3	0.58	17
TCE	14.7	11.8	0.80	20
Octane	54.2	88.1	1.63	33
Toluene	21.5	31.1	1.45	25
Nonane	57.4	242.0	4.22	49
m+p-Xylene	41.6	138.4	3.33	36
o-xylene	11.2	61.2	5.46	39
Decane	184.7	957.6	5.18	70
1,3,5-TMB	9.7	52.6	5.42	50
Undecane	262.8	1939.3	7.38	105
1,2-DCB	27.0	248.4	9.20	43
Dodecane	61.4	1899.0	30.9	155
Total	1090	5839	5.81	

TABLE 13. CUMULATIVE MASS OF TARGET COMPOUNDS REMOVED DURING STEAM INJECTION.

Target Compound	Cumulative Mass Removed During Steam Injection				
	(grams)	(pounds)			
Hexane	24.7	0.05			
cis-1,2-DCE	103.1	0.23			
1,1,1-TCA	57.4	0.13			
Heptane	99.2	0.22			
Benzene	1.6	0.00			
TCE	41.9	0.09			
Octane	122.9	0.27			
Toluene	47.6	0.11			
Nonane	333.8	0.74			
m+p-Xylene	190.5	0.42			
o-xylene	64.8	0.14			
Decane	1290.7	2.84			
1,3,5-TMB	91.6	0.20			
Undecane	2204.5	4.86			
1,2-DCB	480.9	1.06			
Dodecane	916.2	2.02			
Total	6071.3	13.37			

### G. POST-STEAM VAPOR EXTRACTION

After 100 hours the steam injection was terminated while the vapor extraction system was left operating. The continued vapor extraction served two purposes: to remove heat from the test cell and to evaporate residual contamination. The SVE was continued for over 2 weeks after the steam injection ceased. The cell cooling followed an exponential decay as illustrated by the vapor temperatures measured at the extraction wells shown in Figure 54. The majority of the cooling occurred within the first 48 hours of operation. The applied vacuum at the extraction

wells remained relatively constant at 559 mmH<sub>2</sub>O throughout this phase (see Figure 55). The total air extraction rate increased at the start of cooling and then was nearly constant at 1.13 scmm (40 scfm). This is illustrated in Figure 59. As less water vapor was removed, more air was extracted. The evaporation of pore water can also be seen in Figure 57 where the water removed from the cell increased exponentially at the beginning of the cooling. The cooling removed an additional 1,263 liters (334 gallons) of water from the cell. Comparing the air extraction rate during SVE to the air extraction rate during cooling is also an indicator of water removal. The ratio of SVE to cooling air flow is 0.63, which suggests an approximate initial water saturation of 23% if the cooled soil is assumed dry. Displacement of fine particles from pore spaces by steam flow could also have increased the absolute permeability.

Vapor concentrations of 12 of the target compounds measured with the on-site GC during the cell cooling are plotted versus time in Figure 60. The contaminant concentrations dropped off exponentially as the cell cooled. The final vapor concentrations were almost two orders of magnitude less than the final SVE concentrations preceding steam injection.

The masses of target compounds removed during the cell cooling were calculated from the measured extraction rate and the measured concentrations. The majority of the mass was recovered during the initial cooling. These calculated masses are presented below in Table 14. During the cell cooling, 24,260 cubic meters (856,650 cubic feet) of air was extracted from the test cell. This volume yields an average total target compound concentration of 106 mg/m<sup>3</sup>. The total period of extraction in this phase was 356 hours yielding an average target mass removal rate of 7.2 grams (.25 ounce) per hour.

## H. NAPL AND TARGET COMPOUND REMOVAL EFFICIENCY

#### 1. Mass Balance

A mass balance was performed by comparing estimates of contaminant masses in the soil before and after steam injection with estimates of the mass removed in the effluent streams. In addition, several soil samples were analyzed for total petroleum hydrocarbons (TPH). The TPH measurements were combined with the concentrations of target compounds to develop mass fractions of target compounds in the NAPL. The estimated NAPL makeup and mass from these

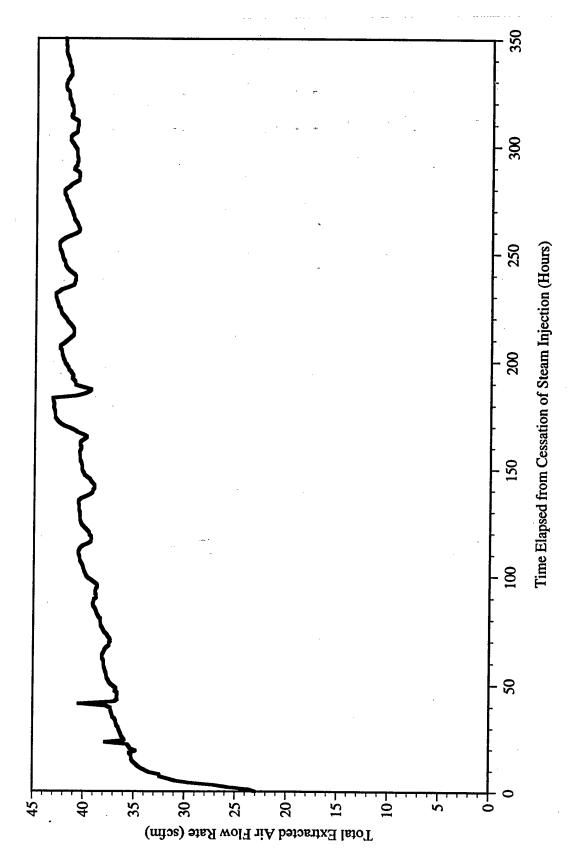


Figure 59. Total Extracted Air Flow Rate After Ceasing Steam Injection.

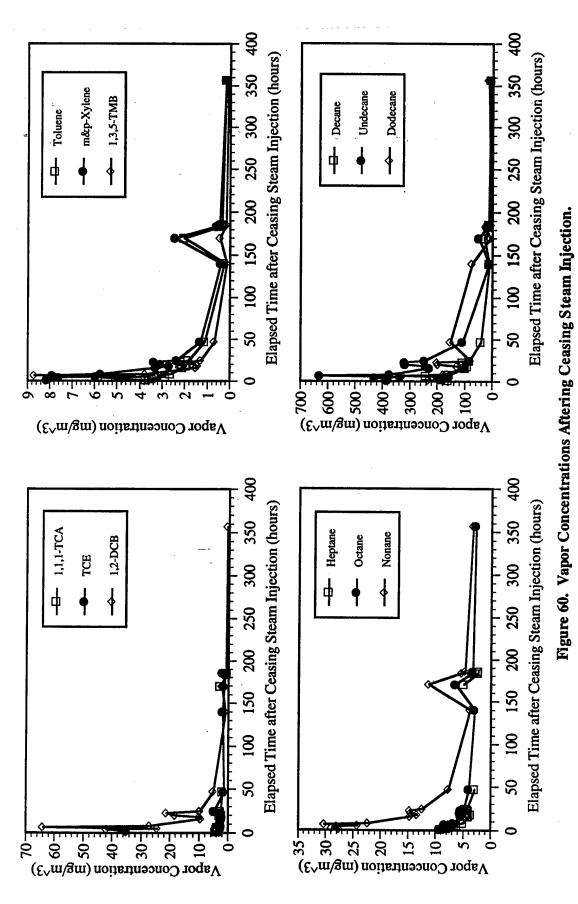


TABLE 14. CUMULATIVE MASS OF TARGET COMPOUNDS REMOVED DURING CELL COOLING.

Target Compound	Cumulative Mass Removed During Cell Cooling		
	(grams)	(pounds)	
Hexane	9.7	0.02	
cis-1,2-DCE	115.2	0.25	
1,1,1-TCA	22.5	0.05	
Heptane	23.5	0.05	
Benzene	0.1	0.00	
TCE	26.6	0.06	
Octane	84.0	0.19	
Toluene	22.1	0.05	
Nonane	128.7	0.28	
m+p-Xylene	17.5	0.04	
o-xylene	14.2	0.03	
Decane	437.4	0.96	
1,3,5-TMB	34.6	0.08	
Undecane	854.0	1.88	
1,2-DCB	239.4	0.53	
Dodecane	544.9	1.20	
Total	2574.5	5.67	

calculations are presented in Table 15. As shown, the target compounds makeup a small portion of the NAPL. Other compounds, in particular heavier alkanes, are expected to makeup the majority of the NAPL. The total mass of contaminants in the test cell was estimated to be 406 kg (896 pounds) which for a NAPL density of 0.75 g/cm3 yields 545 liters (144 gallons). This estimate is higher than predicted by the PITT but the calculation does not account for partitioning in the volatilized, dissolved, and adsorbed phases which would tend to reduce the estimate.

The masses of target compounds in the soil were based on measured soil concentrations and assuming blocks of uniform concentration around point measurements. 89 samples were used to calculate the initial mass and 47 were used for the post-steam mass. Effluent masses were calculated using measured concentrations and flowrates of extracted air and groundwater

TABLE 15. ESTIMATED INITIAL NAPL MAKEUP AND MASS.

Compound	Estimated NAPL Mass Fraction (a)	Molecular Weight	Estimated NAPL Mole Fraction	Estimated Mass Based on Soil Concentrations (grams)	Estimated Mass Based on NAPL Makeup and Soil Concentrations (grams)
Solvents	<i></i>				
cis-1,2-DCI	(b)	96.944	(b)		NA
1,1,1-TCA	(b)	133.405	(b)		#
TCE	(b)	131.389	(b)	1	1.28
1,2-DCB	0.003167	147.004	0.00421	1,439.67	1,286.26
Aromatics					
Benzene	(b)	78.114	(b)	1.21	1.21
Toluene	0.000724	92.141	0.00154	274.43	274.43
Ethylbenze	0.000277	106.168	0.00051	103.47	112.70
m&p-Xylen	0.000566	106.168	0.00104	198.76	229.90
o-Xylene	0.001385	106.168	0.00255	505.49	562.41
1,3,5-TMB	0.000827	120.195	0.00134	308.62	335.73
Naphthalen	0.000505	128.174	0.00077	217.11	205.22
<u>Alkanes</u>					
Hexane	(b)	86.178	(b)	NA	NA
Heptane	0.000077	100.205	0.00015	NA	31.23
Octane	0.000174	114.232	0.00030	NA	70.55
Nonane	0.001010	128.259	0.00154	NA	410.29
Decane	0.007583	142.286	0.01042	3,012.84	3,079.87
Undecane	0.017275	156.313	0.02160	7,016.24	7,016.24
Dodecane	0.012595	170.340	0.01445	NA	5,115.61
Other	0.953835	198.394	0.93959	NA	387,406.44
TOTAL	1.000000		1.00000		406,214.60

NA = Not Analyzed

Sum MW / Mass Frac = 0.005117

Gallons

143.8

<sup>(</sup>a) Mass fractions estimated from soil, groundwater, vapor, and NAPL samples collected before steam injection.

<sup>(</sup>b) These compounds are not expected to be dissolved in the NAPL because of weathering.

along with the separated volume of recovered NAPL. 91 samples were used to calculate the mass removed in the vapor phase while 68 samples were used for the water phase. The masses of contaminants in the separated NAPL were calculated using the estimated mass fractions presented in Table 15. The results of mass balances for 12 of the target compounds are presented in Table 16. The first columns compare estimated masses in the soil before and after steaming. These estimates reveal over 90% removal for volatile compounds, 80 to 90% removal for moderately volatile compounds and 70 to 80% for semi-volatile compounds. Estimates for the target compound mass removal in the effluent streams reveals that over 90% was extracted in the vapor phase. The estimates of masses removed in the effluent streams were consistently lower than the estimates from the changes in soil concentrations but the same order of magnitude was achieved which is surprising given the complexity of the NAPL and geology. The total mass of NAPL removed in the vapor phase was qualitatively estimated by averaging the sum of the areas of the target compounds in the gas chromatography results relative to the total areas of the chromatograms. The calculated average revealed that the target compounds made up roughly 19% of the total mass in each sample. Using this factor, the total mass removed in the vapor phase was estimated to be about 33,000 grams (73 pounds) as indicated in Table 16. Assuming a unit weight of 0.75 g/cm3 for the NAPL, this equates to about 45.5 liters (12 gallons).

Further insight into the impact of the steam injection can be gained by evaluating soil concentrations as a function of depth. The average soil concentrations before and after steam injection for toluene; 1,2-DCB and undecane are plotted versus depth below the surface in Figure 61. These plots are typical for the target compounds and reveal excellent removal from the top of the NAPL-contaminated soil down to a depth of about 6 meters (20 feet). Reviewing Figures 50 to 52 shows that the bottom of the steam zone was at a depth of about 6 m. Therefore, the soils exhibiting excellent cleanup are those which were swept by the steam. The soils which were not swept show reductions but not as profound. The difference in average concentration reductions for the two regions is presented in Table 17. The steam swept soils were cleaned of the target compounds by over 94% including the semi-volatile compounds while reductions in the soils below the steam zone were much less. A deeper steam sweep was not possible in this field test because the groundwater pump inlets could not be placed deeper than

ESTIMATIONS OF MASSES REMOVED FROM THE TEST CELL TABLE 16.

Compound	Estimation	Estimation of Mass Removed Based on Soil Concentrations	sed on Soil Conc	entratione	7.00	200		
	Pre-Steam Mass in the Test Cell (grams) (a)	Post-Steam Mass in the Test Cell (grams)	% Reduction in Pre- and Post- Masses	Estimated Mass Removed (grams)	Pha (S	nation of Mass Remo Mass Removed in Water Phase (grams)	cd Mass Removed Based on Extracted Fluids cd Mass Removed Mass Removed in Estim se in Water Phase Separated NAPL Extra (grams) (grams)	d Fluids  Estimated Mass in  Extracted Fluids  (grams)
					(b)		(P)	34
Solvents								
1,1,1-TCA	75.23		5	73.32	84.74	6.38	(9)	- 10 - 10
1.2 DCB	1.28	3.14		( <del>q</del> )	51.73	6.14		57.87
	0.00	471.19	%17	1,017.89	429.19	40.11	28.63	497.93
Aromatics								
Benzene	1.21	1.22	<b>(</b>	ê	192	A7.0	-	
Toluene	274.43	23.85	6	250.59	7	10.56	<b>e</b> (	2.70
m&p-Xylene	198.76	32.44	84%	166.32		5.01	(6)	26.80
o-Xylene	505.49	93.54	81%	411.95	254.07	12.5	7.00	110.65
1,3,5-TMB	308.62	59.90	81%	248.72	70.93	32.90	1.09	273.31
Naphthalene	217.11	59.82	72%	157.29	66.55	10.07	5.24	81.86
Alkanes							•	
Decane	3,012.84	783.51	74%	2,229.33	1,901.52	33.95	70.82	2.006.30
	1,010.24	1,735.67	75%	5,280.57	3,312.94	80.32	146.86	3,540.12
Total Targets	13,050.90	3,216.79	75%	9,835.98	6,340.86	239.66	285.11	6,865.63
Total NAPL	406,215	•	•		33,107			20 00
							•	701,66

Notes

(a) The estimated total mass of each compound in the cell is calculated using block averages of the soil concentrations presented in Figures 39-42.

(b) The estimated masses of these compounds increased after steaming but most samples were non-detect or just above the detection limit invalidating the estimates.

(c) The total NAPL removed in the vapor phase was estimated by multiplying the total target compound mass by 5.2, an estimated response factor (d) The estimated mass of each compound in the NAPL was calculated using the estimated mass fraction of each compound multiplied by for the total contaminant mass in vapor samples based on the ratio of target areas to total area detected.

the measured separated NAPL of 9.5 liters (6,800 grams).

(e) These compounds are not expected to be dissolved in the NAPL because of weathering.

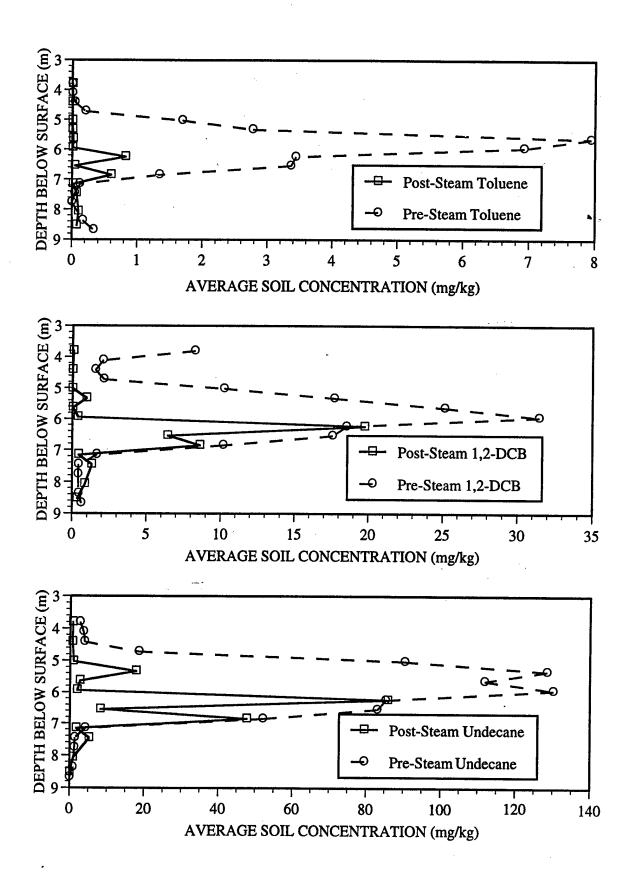


Figure 61. Comparison of Pre-Steam and Post-Steam Soil Concentrations Versus Depth.

TABLE 17. COMPARISON OF AVERAGE PRESTEAM AND POSTSTEAM SOIL CONCENTRATIONS.

Compound	Average Soil Concentrations from Depths 4.2 to 6.1 m (Steam Zone)			Average Soil Concentrations from Depths 6.1 to 7.3 m (Below Steam Zone)			
	Pre-Steam	Post-Steam	% Reduction	Pre-Steam	Post-Steam	% Reduction	
1,1,1-TCA	0.555	0.001	99.7%	1.102	0.053	95.2%	
TCE	0.013	0.031	(a)	0.015	0.023	(a)	
1,2-DCB	14.701	0.287	98.1%	11.992	8.812	26.5%	
Benzene	0.007	0.007	3.7%	0.010	0.006	36.7%	
Toluene	3.264	0.011	99.7%	2.064	0.372	82.0%	
Ethylbenzene	1.251	0.007	99.4%	0.762	0.416	45.5%	
m&p-Xylenes	2.582	0.019	99.3%	1.253	0.684	45.4%	
o-Xylene	6.239	0.068	98.9%	3.644	1.785	51.0%	
1,3,5-TMB	3.927	0.074	98.1%	2.026	1.374	32.2%	
Naphthalene	2.296	0.119	94.8%	1.980	1.167	41.0%	
Decane	35.018	1.218	96.5%	24.776	16.425	33.7%	
Undecane	80.594	4.807	94.0%	56.148	35.912	36.0%	
Total	150.448	6.649	95.6%	105.772	67.012	36.6%	

<sup>(</sup>a) The TCE concentrations could not have increased; many samples were non-detect.

6 m. It is expected the same high levels of removal would have been achieved in the lower soils if deeper screen and pump placement had been possible.

#### 2. NAPL Distillation

As described above, the primary mechanism for contaminant removal was distillation into the flowing vapor. This process is similar to the evaporation which occurs during soil vapor extraction and the results from SVE and steaming can be almost directly compared to determine the effectiveness of the steam enhancement. To facilitate modeling of the process, the soil inside the test cell was considered to be a single lump (i.e., a well-stirred mixture). This approach is

valid after steam breakthrough when transients in the test cell are relatively slow. To compare SVE and steam, a lumped recovery efficiency (ηi) is defined as the measured vapor concentration of compound i at the extraction manifold divided by its saturated, pure-component vapor concentration at the system temperature:

$$\eta_{i} = \frac{C_{\text{measured},i}}{C_{\text{sat},i}} \tag{7}$$

and

$$C_{\text{sat,i}} = \frac{P_{\text{v,i}} M_{\text{i}}}{R_{\text{u}} T} \tag{8}$$

where Pv,i is the pure component vapor pressure at the system absolute temperature T, Mi is the component molecular weight, and Ru is the universal gas constant. This definition compares the measured vapor concentration with the theoretical maximum concentration which would result from equilibrium between the flowing vapor and a pure liquid. This approach lumps the mass transfer constraint of dissolution in the NAPL with other constraints and soil heterogeneities. If the NAPL makeup were known, the saturated concentration could be multiplied by the mole fraction of the component to separate out this constraint. In this study, the NAPL is weathered and the composition of the NAPL changes during the test so that the mixture constraint is included in a single system efficiency. The definition of the saturated concentration reveals it to be a strong function of the system temperature since the component vapor pressure is a strong function of temperature. For the comparison of SVE and steam distillation, the SVE is assumed to occur at 15 °C and the steam distillation at 90 °C.

The thermodynamic properties of the target compounds are presented in Table 18. The table includes vapor pressure at ambient soil temperature (15°C), water solubility, Henry's constant, and calculated saturated vapor concentrations at 15 °C and 90 °C. The final column lists the ratio of steam to ambient vapor concentrations providing an indicator of the concentration increase over SVE expected under ideal steam injection conditions. Volatile compounds are expected to increase by one order of magnitude while semi-volatile compounds may increase by two orders. Pseudo-steady vapor concentrations are listed for SVE and steam

TABLE 18. THERMODYNAMIC PROPERTIES OF TARGET COMPOUNDS.

Compound	Vapor Pressure at 15 °C (kPa)	Solubility in in Water at 20°C (mg/L)	Henry's Constant (kPa m^3/mol)	Saturated Concentration at 15 °C (mg/m^3)	Saturated Concentration at 90 °C (mg/m^3)	Concentration Ratio 90°C / 15 °C
Salvente		·				
Solvents cis-1,2-DCE	17.345	3500.0000	0.768	701 000	7 000 547	
1,1,1-TCA	10.408		2.800	701,882	7,822,547	11
TCE	4.957	1100.0000	0.923	7	7,136,913	12
1,2-DCB	0.199	145.0000	'	271,882	4,814,796	18
1,2-DCB	0.199	143.0000	0.190	12,181	348,986	29
Aromatics					:	
Benzene	7.824	1780.0000	0.550	255,099	3,513,704	14
Toluene	1.821	515.0000	0.670	70,026	1,649,862	24
Ethylbenzene	0.559	152.0000	0.800	24,777	850,537	34
m&p-Xylenes	0.452	162.0000	0.710	20,043	769,820	38
o-Xylene	0.364	175.0000	0.500	16,130	650,355	40
1,3,5-TMB	0.109	97.0000	0.370	5,469	363,611	66
Naphthalene	0.012	34.4000	0.043	621	53,599	86
Alkanes						
Hexane	12.823	10.0000	200.0	461,256	5,383,377	12
Heptane	3.608	2.9300	230.0	150,931	2,606,100	17
Octane	1.028	0.6600	300.0	49,025	1,268,053	26
Nonane	0.221	0.1220	500.0	11,809	609,277	52
Decane	0.061	0.0520	700.0	3,642	245,672	67
Undecane	0.017	0.0440	1900.0	1,125	144,578	128
Dodecane	5.17E-03	0.0034	786.0	367	70,185	191
Tridecane	1.46E-03			112	25,378	226
Tetradecane	8.52E-04		110.0	70.55	16,630	236
Pentadecane	1.21E-04		İ	10.75	5,512	513
Hexadecane	4.45E-05			4.20	2,886	687
Heptadecane	1.39E-05		[	1.40	1,406	1,005
Octadecane	7.84E-06		Į.	0.833	926	1,112
Nonadecane	2.36E-06			0.264	444	1,681
Eicosane	4.23E-07			0.050	158	3,162

and the first sample during steam injection which can be observed in Figures 50 and 59. The pseudo-steady steam concentrations are averages over the period from 50 to 100 hours of steam injection. The fourth column of Table 19 lists the ratios of the steam to SVE average concentrations. When this ratio is compared to the ratio of saturated concentrations listed in the last column of Table 18, it is apparent the steam heating did not increase the recovery rate as much as the increase in volatility indicated. Further, the vapor sampling procedure during steam injection followed these steps: draw off a sample in a gas tight syringe, bring the sample to ambient temperature (the steam condensed), equilibrate the gas in the syringe to ambient pressure, and subsample the air in the syringe. The measured air concentration must then be corrected to an apparent steam vapor concentration by calculating the mass ratio of steam to air in the original sample and accounting for contaminant mass in the condensed steam which was not measured. The results of this correction are listed as the steam corrected vapor concentration in Table 19. The corrected concentrations are lower than the measured air concentrations because the air content in the original vapor samples was less than the steam vapor content.

Comparison of the vapor concentrations before and during steam injection provides insight into the mechanisms of the contaminant recovery as presented in Figure 62. The ratios of the steam to SVE average concentrations show the most volatile compounds did not see a significant increase in concentration (ratio ~ 1). For compounds with ambient vapor pressures higher than 1 kPa, the steam had no impact on recovery. This can be explained by weathering of the NAPL. Over time the more volatile compounds have partitioned out of the NAPL (if these compounds were ever in the original NAPL) into groundwater and air. The general trend in Figure 62 is for an increasing concentration ratio for decreasing saturated concentration with two exceptions showing greater increases. The 1,2-DCB and 1,3,5-TMB both exhibited greater increases than the general trend predicts. A plausible explanation for this observation is the high solubility of these compounds in water compared to the alkanes in the NAPL. Liquid water is present in the steam zone and can inhibit distillation if it exists between the NAPL and steam vapor. This effect is lessened the more soluble a compound is in water. Also, the steam passed over a portion of the NAPL-contaminated zone and these compounds may have been more

TABLE 19. REMOVAL EFFICIENCIES FOR VAPOR EXTRACTION DURING EACH PHASE.

Ratio of Cool Efficiency to Steam Efficiency	3.84 2.44 2.29	2.01 2.31 1.06 2.90	1.90 1.89 2.25 3.79 5.35 14.16
Ratio of SVE Efficiency to Steam Efficiency	42.85 56.78 104.50 14.16	114.51 92.78 104.06 65.37	52.26 85.69 99.05 111.38 161.43 205.63
Cooling Efficiency (d)	0.000286% 0.000063% 0.000067% 0.011111%	0.000238% 0.000800% 0.001429% 0.001538% 0.100000%	0.000333% 0.000833% 0.005882% 0.100000% 0.400000% 0.500000%
Steam Efficiency	0.000074% 0.000026% 0.000020% 0.004852%	0.000118% 0.000346% 0.001342% 0.000531%	0.000022% 0.000175% 0.000440% 0.002609% 0.074813% 0.035316%
SVE Efficiency	0.003192% 0.001457% 0.002051% 0.068687%	0.013568% 0.032098% 0.139611% 0.034694%	0.001160% 0.015035% 0.043582% 0.307883% 2.939448% 12.076649% 7.261960%
Steam Conceted Vapor Concentration (mg/m^3) (c)	5.83 1.83 0.94 16.93	1.95 2.66 8.73 1.93	1.20 4.57 5.58 15.90 64.83 108.16 24.79
Ratio of Steam Conc to SVE Conc	1.38 1.16 0.90 10.50	1.09 2.19 2.05 5.35	1.19 1.08 1.39 2.33 3.23 4.25 4.96
Steam Steady Vapor Concentration (mg/m^3) (b)	30.9 9.8 5.0 87.9	10.4 14.1 46.1 10.2	6.4 24.4 29.8 84.9 346.1 577.3 132.3
SVE Steady Vapor Concentration (mg/m^3) (a)	22.4 8.4 5.6 8.4	9.5 6.4 22.5 1.9	5.4 22.7 21.4 36.4 107.1 135.9 26.7
Compound	Solvents cis-1,2-DCE 1,1,1-TCA TCE 1,2-DCB	Aronaucs Toluene m&p-Xylenes o-Xylene 1,3,5-TMB	Alkanes Hexane Heptane Octane Nonane Decane Undecane

(a) The steady SVE concentrations are the averages from the last two SVE samples and the first steam sample. Notes:

<sup>(</sup>b) The steady steam concentrations are the averages from 50 to 100 hours after steam injection started.

<sup>(</sup>c) The corrected steam concentration represents the apparent vapor concentration in the steam vapor only as derived from the measured air concentration.

<sup>(</sup>d) The cooling efficiency represents a best fit to the vapor concentration data while the test cell cooled.

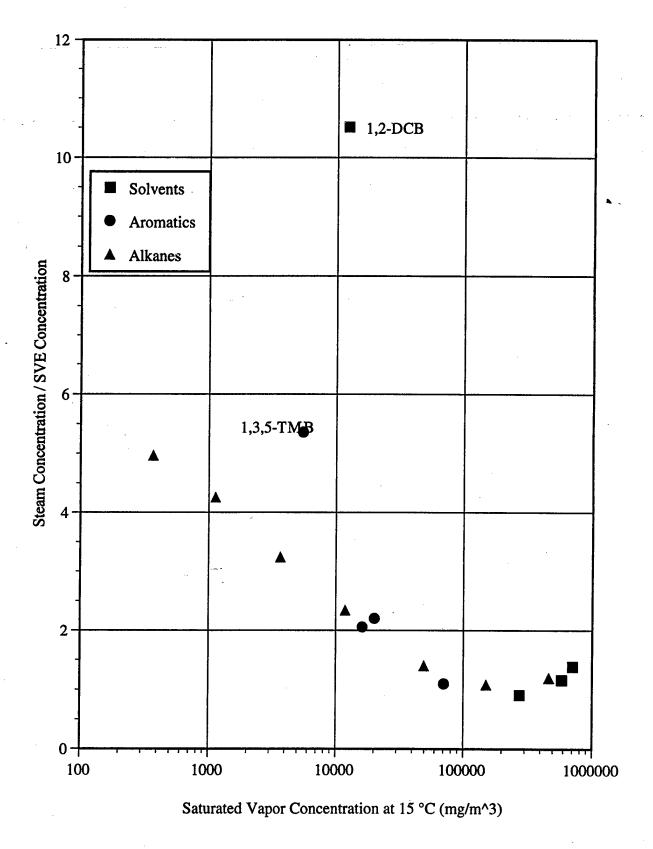


Figure 62. Steam to SVE Concentration Ratios as a Function of Ambient Saturated Concentration.

readily volatilized because of their solubility. The solubility of these compounds is roughly 3 orders of magnitude higher than the alkanes.

The removal efficiencies of SVE and steam injection as defined above are both shown in Table 19. The steam efficiency was calculated using the corrected concentration. The efficiency during the vapor extraction accompanying the cell cooling is also listed. The cooling phase efficiency was determined by using the average temperature of the test cell and plotting a best fit curve of the calculated saturated concentration at the cell temperature multiplied by the efficiency simultaneously with the measured vapor concentrations. The decay in vapor concentrations illustrated in Figure 60 followed closely the decay in temperature within the test cell. The removal efficiency at the end of the SVE phase was roughly two orders of magnitude better than during steady steam injection as suggested by the relatively modest increase in concentrations during steam injection. The efficiency during the cooling phase was also higher than during the steam phase.

Two possible explanations for the low efficiency during the steam injection are: (1) the introduction of water as steam may "encapsulate" the non-wetting NAPL providing a liquid/liquid diffusion barrier to evaporation into the vapor phase, and (2) an increased relative permeability in the NAPL zone of the three-phase system which increases the by-pass of vapor flow. The water encapsulation is the result of liquid from condensing steam which provides the energy for distillation; a balance must exist between the rate of steam condensing and the rate of NAPL distillation - the question becomes, how much does the liquid water inhibit the distillation? Also, capillary condensation in the interstitial pore space can bring liquid water to the NAPL zone.

# SECTION V CONCLUSIONS

#### A. PRETREATMENT PITT

### 1. Method of Moments Analysis

The data set from the pretreatment PITT that was provided to INTERA by Applied Research Associates, Inc. was analyzed using the Method of Moments technique. Data analysis results indicate that about 469 liters (124 gallons) of NAPL are present in the saturated zone of the test cell. The NAPL is nonuniformly distributed in the test cell with NAPL saturation ranging from 1 percent to 8 percent. The average NAPL saturation over the entire saturated zone is approximately 5 percent.

## 2. Inverse Modeling Technique

The inverse modeling technique was used as a second method to determine an estimation of the spatial distribution of the hydraulic conductivity and NAPL saturation in the test cell. The analysis was performed on the same data set that was used above for the Method of Moments Analysis. The hydraulic conductivity field was obtained by the history matching of the field measurement of methanol data. The NAPL saturation was obtained by using both methanol and 2,2-dimethyl, 3-pentanol data. In general, the results from the method of inverse modeling are in good agreement with the results from the method of first temporal moment analysis. The results from the inverse modeling indicate that there are approximately 393 liters (104 gallons) of NAPL present in the pore space between the rows of injection and extraction wells. The NAPL is non-uniformly distributed in the test cell, ranging from 0 percent to 10 percent in saturation. The average NAPL saturation in the test cell is approximately 5 percent.

### B. STEAM INJECTION/VAPOR EXTRACTION

In the previous sections, the results of the steam treatability study were described in three major phases. The first phase consisted of dewatering the test cell and performing SVE at ambient temperature to provide a baseline for comparing the recovery enhancement from steam injection. The second phase was steam injection with dual phase extraction. The third phase

consisted of cell cooling by continuing SVE after ceasing steam injection. The conclusions from each phase are presented below.

The vapor concentrations of the more volatile compounds such as 1,1,1-trichloroethane (1,1,1-TCA) and heptane during the initial ambient SVE were initially high and exhibited the exponential decay characteristic of long-term SVE. The concentrations of these compounds dropped about 80 percent during this short period of testing. For moderately volatile compounds such as toluene and nonane, the vapor concentrations appeared to decrease slightly during the tests. Concentrations of compounds with relatively low volatility, such as 1,2-dichlorobenzene (1,2-DCB) and undecane, were erratic and did not appear to decrease during the SVE testing. A cumulative mass of 903.5 grams (1.99 pounds) of the target compounds were extracted during the initial SVE at an average rate of 19 grams (.67 ounce) per hour and an average vapor concentration of 445 mg/m<sup>3</sup>.

Steam injection was initiated to mobilize NAPL and evaporate contaminants. The initial steam flow was preferential toward the southeast extraction well (U2-2741). Steam reached Extraction Well 41 after approximately 11 hours of injection and well U1-2744 after about 14 hours. The steam zone reached Extraction Wells U1-2751 and U1-2753 after about 20 and 21 hours, respectively. Shortly after steam breakthrough in all four extraction wells, the flow became effectively steady with the steam extracted nearly equaling the steam injected. This preferential flow could not have been predicted from the several, closely spaced geologic logs from the cell.

A bank of NAPL preceding steam breakthrough was considered possible; yet, only about 9.5 liters (2.5 gallons) of NAPL were recovered in the NAPL/water separator after steam breakthrough. This indicates the steam injection was not effective at driving the residual NAPL out of the cell. This occurred because the viscosity of the NAPL was too high and the saturation too low to allow the formation of a stable NAPL bank ahead of the steam condensation front. Theory predicting the maximum NAPL viscosity which allows a stable NAPL bank to mobilize was developed and suggested the maximum NAPL viscosity allowing stable displacement by steam injection at OU-1 is about 2.5 centipoise (cP). The NAPL at Operable Unit One has a

viscosity significantly higher than 2.5 cP. Therefore, the primary recovery mechanism was distillation of the NAPL components.

The measured vapor concentrations of target compounds during the first 10 hours of steam injection, before steam breakthrough, showed no significant change from the SVE concentrations. Even after steam breakthrough, the most volatile of the target compounds (e.g., 1,1,1-TCA; heptane; TCE) did not increase significantly in concentration with the temperature\* increase. This indicates these compounds are dissolved in a NAPL or water that is not being heated. In fact, the peak concentrations of the volatile compounds during early, ambient SVE were higher than the maximum concentrations detected during steam injection. This comparison of maximum concentrations also indicates disequilibrium within the NAPL (i.e., a nonuniform mixture) that would result from weathering of the NAPL over time or the presence of two distinct NAPL layers. The site history indicates two NAPL layers: one NAPL was the result of hydrocarbon usage for fire training, while the other NAPL resulted from chemical disposal pits that included the release of solvents. The concentrations of moderately volatile compounds such as octane, toluene, nonane, and xylenes showed a significant increase with the increase in temperature, but not nearly as much as would be predicted by the increase in their vapor pressures. The weathering and disequilibrium are expected to be at a lesser degree than the volatile compounds as was observed in the data. For the semi-volatile compounds such as 1,2-DCB, undecane, and dodecane the vapor concentrations were much increased during steam breakthrough, although the concentrations dropped to apparent steady state values after steam breakthrough. A cumulative target compound mass of 6,071 grams (13.4 pounds) was removed during the steam injection phase. The average total target compound concentration during steam injection was 1,900 mg/m³ in the air (neglecting the steam vapor). The total period of steam injection and extraction was 100 hours yielding an average target mass removal rate of 60.7 grams (2.14 ounces) per hour.

After 100 hours, the steam injection was terminated while the vapor extraction system was left operating. The continued vapor extraction served two purposes: to remove heat from the test cell and to evaporate residual contamination. The SVE was continued for over 2 weeks after the steam injection ceased. The cell temperature followed an exponential decay and the majority

of the cooling occurred within the first 48 hours of operation. The target compound concentrations also dropped exponentially as the cell cooled. The final vapor concentrations were almost two orders of magnitude less than the final SVE concentrations preceding steam injection.

The majority of the mass of target compounds removed during the cell cooling were recovered during the initial cooling. The average total target compound concentration was 106 mg/m³ at an average target mass removal rate of 7.23 grams (.25 ounce) per hour. A cumulative target compound mass of 2,575 grams (5.7 pounds) was removed during the post-steam injection SVE phase.

The final soil and groundwater concentrations in the test cell were significantly reduced from the pre-test concentrations. Estimates of mass removed based on soil concentrations before and after steaming reveal over 90% removal for volatile compounds, 80 to 90% removal for moderately volatile compounds and 70 to 80% for semi-volatile compounds. In addition, soils swept directly by the steam exhibited excellent cleanup and the soils which were not swept showed reductions but not as profound. The steam swept soils were cleaned of the target compounds by over 94% including the semi-volatile compounds. A deeper steam sweep was not possible in this field test because the groundwater pump inlets could not be placed deeper than 6 m. It is expected the same high levels of removal would have been achieved in the lower soils if deeper screen and pump placement had been possible.

Approximately 34 kg (75 pounds) of NAPL were removed from the cell through the vapor stream during the course of the experiment. Assuming a unit weight of 0.75 g/cm3 for the NAPL, this equates to about 45.5 liters (12 gallons). An additional 9.5 liters (2.5 gallons) were recovered in the NAPL/water separator.

#### C. POSTTREATMENT PITT

ARA is currently awaiting the results of the data analysis from the posttreatment PITT being conducted by INTERA, Inc. A copy of the results and a discussion comparing the pre- and posttreatment PITT results will follow as an addendum upon receipt.

# SECTION VI RECOMMENDATIONS

Based upon the results obtained during the course of this project several recommendations can be made. The results from this study showed steam injection to be very effective in distilling contaminants from the mixed NAPL at OU-1, Hill AFB. Yet, the increase in vapor concentrations of the moderately volatile compounds was not as high as expected based upon the vapor pressures of these compounds at the elevated temperature. Further study is warranted to evaluate the reasons for this lack of increase. In particular, the role which liquid water may play in this process needs more investigation because further understanding could lead to substantial improvements in the technology implementation. Additionally, a substantial bank of NAPL was not pushed ahead of the steam condensation front in this demonstration. Theory was presented suggesting a relatively low limiting viscosity for such a push to occur. This theory requires additional laboratory and field studies for validation because of the potential impact this result could have on how the technology is applied to heavier hydrocarbons. Also, the evaluation of the technology for other contaminants and soil types should be pursued.

Any of these additional studies should also have comments as to the costs of using steam injection as a remediation technology. For coarse, gravely soils, such as those at Hill, the injection and extraction wells can be relatively far apart; whereas for fine grained soils, more wells per unit area may be required, driving the cost higher than experienced during this study. These costs need to be considered when performing a complete evaluation of steam injection remediation for a site. In addition, the feasibility of using pushed wells for injection and extraction of the steam should be studied. This well installation procedure has the potential to be faster, cheaper, and more informative without any loss in performance.

A second recommendation is to further enhance and develop the Partitioning Interwell
Tracer Test (PITT). Although this test was very useful in determining the pre- and postcontaminant locations and saturation levels, performing the test was relatively expensive and
very labor intensive. Additional methods to reduce the costs of performing these tests would
greatly assist in increasing the utility of these tests. A new approach would still use partitioning

tracers, but rather than collect samples over a 10-day period (over 2,000 samples were collected and analyzed for each PITT test during this demonstration), a monitoring system could be used to monitor the partitioning in-situ. This would require a sensor network to be installed and different tracers to be selected that matched with the sensing technology chosen for the network. One sensing technology that should be investigated is fluorescence techniques. If partitioning fluorescence sensors can be selected, then a network of simple fluorescence probes could be used to monitor the experiment. Since the sample collection rate would not be limited by actual sample collection time, more detailed results can be collected at low additional cost. A Cone Penetrometer fluorescence sensor version can be used to monitor the tracers in an open field condition under a lower gradient. These approaches effectively reduce costs and allow more flexibility in the tracer flow field.

Once the PITT data has been collected, additional detailed analysis is needed to better understand the permeability and NAPL saturation distributions. Two numerical approaches have been presented but additional graphical display and numerical modeling to ensure the flow situations are correct should be performed. The PITT provides excellent insight into the site specific flow field conditions as well as where the pockets of contamination are located. By using this information, the remediation techniques can be efficiently focused to ensure a cost effective solution to the contamination problem.

Finally, it is recommended that an additional study very similar to this study, but at a different site, be performed to confirm the results that were obtained for the effectiveness of the steam injection remediation techniques at this site. This type of testing would allow a different soil type and contaminant mix to be evaluated. In addition, this test should be conducted in a free field configuration to eliminate unique characteristics due to the cell configuration.

# SECTION VII

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# APPENDIX A

# **RSKSOP-72**

STANDARD OPERATING PROCEDURE QUANTITATIVE JP-4 JET FUEL IN COARSE AND MEDIUM TEXTURED SOILS BY GAS CHROMATOGRAPHY

# STANDARD OPERATING PROCEDURE QUANTITATIVE JP-4 JET FUEL IN COARSE AND MEDIUM TEXTURED SOILS BY GAS CHROMATOGRAPHY

RSKSOP-72 Revision No. 1.1 Date: 07/25/95

H3.0.1. Disclaimer: This is a revised Standard Operating Procedure that originally was prepared for use by the Robert S. Kerr Environmental Research Laboratory of the USEPA and may not be specifically applicable to the activities of other organizations.

#### **H3.1 SCOPE AND APPLICATION**

H3.1.1. This method is applicable to the quantitative analysis of aviation gasoline (AVGAS), JP-4 jet fuel, and select individual compounds of the fuels in coarse and medium textured soils. Although not determined when developing this method, these fuels can be quantified in water by this procedure (ref. 3.2). The m- & p-xylenes co-elute on the column used in this procedure. Fuel carbon values may be less accurate if fuel has been subjected to weathering. Approximately 10 analytical runs can be performed/8 hour day. The use of an autoinjector allows for unattended operation and overnight analyses. This method is restricted to use by or under the supervision of the analysts experienced in the use of gas chromatography and in the interpretation of chromatography.

#### H3.2 SUMMARY OF METHOD

A soil sample is added to a VOA vial and extracted with methylene chloride and water. The methylene chloride extracts are analyzed by capillary column gas chromatography-flame ionization detector and/or MS. Based on a 15 g sample, the concentration range investigated is approximately 15-15,000 mg/kg for total fuel and 0.2-200 mg/kg for individual compounds.

#### **H3.3 REFERENCES**

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#### **H3.4 PROCEDURE**

### **H3.4.1.** Sample Vial Preparation

H3.4.1.1. Prepare 40 ml VOA vials for sub-samples of subsurface material in the laboratory. Put labeling tape around vials and weigh. Add 5 ml of organic-free water (Milli-Q water) acidified with H2SO4 (pH<2). Reweigh the vial and water along with screw cap and Teflon-lined septum. Add 5 ml GC/GC-MS grade methylene chloride with a glass 5 ml syringe, Teflon tipped barrel, cap, and reweigh.

#### H3.4.2. Sampling in the Field

H3.4.2.1. Jars for collection of core samples will be pre-washed, assembled with lid liners, and labeled in the laboratory. Core samples will be placed in pint (6' of core material) or quart (12' of core material) jars with aluminum-lined lids, and sample identification will be recorded. Core samples will be taken to an on-site trailer and subsampled for extraction. Subsamples of each core-subsection will be placed in the pre-prepared VOA vials containing the extracting solvent. Immediately prior to addition of soil to the VOA vials, reweigh VOA vials, record weights, and compare to original vial weights with solutions added. If the weights are different by more than 0.1 g, the vials are not used. Take a 10 to 15 gram sub-sample from the core sample jar with a stainless steel spatula. Reweigh VOA

vials with soil, and record sample weight and sample identification. Shake samples rigorously for a few minutes and place in ice chest.

#### H3.4.3. Extraction and Analysis

H3.4.3.1. Allow soils to remain in contact with methylene chloride/water extracting solution for approximately 2 days prior to analysis. Maximum holding time 14 days. Secure the vial to a wrist action shaker and shake the sample for 10 minutes. Remove sample and allow phases to separate. (If required, sonicate the sample for about one minute to break up any emulsion present, or freeze sample to separate phases.) Using a 1 ml glass syringe, Teflon tipped barrel, remove approximately 1 ml of the methylene chloride and transfer it to the head of a drying column prepared from a Pasteur pipette containing approximately 2 inches of fired (400° C 4 hrs) sodium sulfate above a glass wool plug. Allow the dried extract to drain into a 2 ml septum vial. Prepare a method blank every 20 samples, or 1 per set of samples received if sample set is less than 20, by extracting 5 ml of organic free water with 5 ml methylene chloride following the same procedure.

H3.4.3.2. In order to obtain soil moisture content, decant remaining solution to wastes and allow vials with extracted soils to air-dry followed by drying in an oven at 105° C for 24 hours. Reweigh vials with soil (no caps). Moisture contents may also be determined by weighing a subsample from the core sample jar onto a pre-weighed aluminum weighing pan and drying in oven at 105 C for 24 hours.

#### H3.4.4. Sample Analysis

H3.4.4.1. Sample analysis is done using a gas chromatograph (GC) with either FID and/or MS analysis. Data acquisition and processing by a chromatographic software package. See Appendix I-4 for the GC method and MS analysis. For FID analysis, quantitation is based on a 4 point, external standard curve. Calibration standards containing the individual compounds are prepared in methylene chloride from individual primary neat compounds. Using the following equation to calculate compound volumes, a 1000 μg/ml primary mixture made up to 50 ml is prepared by adding appropriate volumes of each compound.

$$V_{\text{orig}} = [(C_{\text{soln}}/\text{density})*(V_{\text{soln}})]*(1000)$$
 (1)

 $V_{orig}$  = volume of neat compound which will be added to the 50 ml volumetric flask ( $\mu$ L)

 $C_{soln}$  = desired final concentration of individual neat compound (1 g/ml = 1000  $\mu$ g/ml)

density = mass of the neat compound in grams divided by volume of the compound in 1 ml (g/ml)

 $V_{soln}$  = final volume (50 ml) of methylene chloride solution

1000 = factor to convert milliliters to microliters

H3.4.4.2. Appropriate dilutions are made from this primary dilution to prepare the remaining stock standards at 100-10-1  $\mu$ g/ml. Retention times are determined for each individual analyte. A four point, external calibration curve (1-10-100-1000  $\mu$ g/ml) is established for each individual analyte.

H3.4.4.3. The original concentration of the soil core is calculated using the following equations:

$$C_{\text{orig}} = (C_{\text{soln}})(V_{\text{ext}})/M_{\text{s}}$$
 (2)

$$V_{\text{ext}} = (V_i) - (S_w V_w / dMC)$$
 (3)

where:

Corig = the concentration (mg/kg) of the analyte in the original core.

C<sub>soln</sub> = the concentration in the actual dilution analyzed via GC (mg/L).

 $M_s$  = dry weight of sample (g)

V<sub>i</sub> = initial methylene chloride (ml) added to soil.

Vext = methylene chloride added to sample after equilibration with aqueous phase (ml).

S<sub>w</sub> = aqueous solubility of methylene chloride (34,000 mg/L at 25° C)

 $V_w$  = added water plus residual water from soil core (ml).

dMC = density of methylene chloride (g/L)

# H3.5 MISCELLANEOUS

H3.5.0.1. The analytes in this method are extremely volatile. Care should be taken to minimize losses.

## **H3.6 PRECAUTIONS**

H3.6.0.1. When working with methylene chloride and/or preparing standards from neat compounds it is advisable to work inside a fume hood and to wear protective gloves. Safe laboratory practice is advised.

# Volatile and Semivolatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique

### 1.0 Scope and Application:

Analytical Method used for soil core samples from Hill AFB shipped in 40 ml vials with pH adjusted water and methylene chloride. Alterations to these core samples such as the use of surfactants may require changes to be made to this method.

#### 2.0 Summary of Method:

Upon receipt of samples the vials are stored at 4°c until ready for analyses. Samples are removed from the refrigerator allowed to equilibrate with room temperature and then sonicated for 15 minutes. After sonication the samples are centrifuged and an aliquot of the methylene chloride phase transferred to a 1.8 ml autosampler vial, internal standard added, and is ready for GC/MS analyses.

#### 3.0 Interferences:

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. If possible these interferences must be eliminated or reported.

contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover it is helpful to rinse the syringe used for injection into the GC/MS numerous times before and after injection. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of methylene chloride to check for cross contamination. Since these samples tend to have a highly concentrated background, blanks need to be analyzed as frequently as possible.

#### 4.0 Apparatus and Materials:

Gas ChromatographMass SpectrometerAutomatic SamplerData StationColumn
SyringeBalanceBottles
Hewlett Packard 5890 with capillary injection
Either a Hewlett Packard 5970 or 5972

Hewlett Packard ALS 7673

Hewlett Packard Chemstation
J&W Scientific DB-1701, 30 meters, .25 mm

I.D., .26 um film thickness

10 ul

Analytical, 0.0001 g

Glass with Teflon-lined screw caps or crimp
tops

#### 5.0 Reagents:

Water- Millipore Corporation Milli-Q UV Plus Methylene Chloride- Aldrich Cat. # 32,399-3 99.9+% PRA Grade Organic Analytes- 97% pure or greater
Calibration Standards and Stocks- prepared according to MTU-EEL
Procedure entitled Standard
Preparation Procedure 5/19/92

Internal Standard Tetrachloroethene
MS Tuning Standard Perfluorotributylamine

# 6.0 Sample Collection, Preservation, and Handling:

This is to be performed by others, method unknown. Once samples are received at MTU-EEL they are stored at 4°C for not more than 14 days.

#### 7.0 Procedure:

#### 7.1 Sample Preparation:

In attempt to insure complete extraction from the soil/water matrix into the methylene chloride all samples are sonicated, at room temperature, in our sonicator at 21 kilocycles per second for 15 minutes. After sonication the samples are centrifuged at 2300 revolutions per minutes for 15 minutes to separate the phases. After phase separation a 1.0 ml aliquot is removed from the vial and put into an HP autosampler vial with a Teflon lined septum and crimp cap.

# 7.2 Operating Conditions for the HP 5890 GC:

250°c Injection port-Oven Temp Initial Time-3 minutes 35°c Oven Temp Initial Value-Oven Temp Program Rate-6°C 0 minutes Oven Temp Final Time-Oven Temp Final Value-137°c 280°C Transfer Line Temp-Column Head Pressure-. 8.5 psi Approximate Column Flow Rate- .9 ml/minute 8.0 ml/minute Septum Purge Flow Rate-31.3 ml/minute Split Vent Flow Rate-.5 minutes Splitless Time-

# 7.3 Operating Conditions of the HP ALS 7673:

Sample Wash-	3
Sample Pumps-	3
Sample Volume-	2 ul
Viscosity Delay-	2 seconds
Solvent A (methylene chloride) Wash-	<b>3</b> .
Solvent B (methylene chloride) Wash-	.3

# 7.4 Operation Conditions of the HP Mass Selective Detector:

Electron Multiplier Voltage (Relative to Autotune) - 200 Selective Ion Monitoring (SIM) Mode

Compound	SIM	Start Time	Cycles/Sec
1,1,1-trichloroethane	97.0	2.3	2.80
benzene	78.0	2.3	2.80
trichloroethene	130.0	2.3	2.80
toluene	91.0	4.0	7.63
tetrachloroethene (I.S.)	166.0	5.0	4.10
ethylbenzene	91.0	5.0	4.10
o-xylene	91.0	5.0	4.10
decane	142.0	9.5	2.80
1,3,5-trimethylbenzene	105.0	9.5	2.80
undecane	156.0	11.5	2.80
dichlorobenzene	146.0	11.5	2.80
naphthalene	128.0	14.2	7.63

Average Pressure- 2.6 X 10<sup>-5</sup>

#### 8.0 Calibration:

A seven point calibration curve must first be analyzed to verify proper operation of the GC/MS. After fitting the standards all analytes must meet acceptance criteria. This criteria includes:

- 1. percent relative standard deviations of less than 15 percent for the first seven compounds in section 8.1, 25 percent for naphthalene and o-dichlorobenzene, and 35 percent for undecane. This is used to determine if the GC/MS is exhibiting a deterioration of response (as determined in SW-846 Method 8270B section 7.3.4.1 page 13),
- deviation of any single concentration point greater than;

35 percent for naphthalene and benzene,

1.32

- 20 percent for 1,3,5-trimethylbenzene, undecane, and ethylbenzene,
- 35 percent for the lowest standard of 1,1,1-trichloroethane, trichloroethene, toluene, o-xylene, and decane and 20 percent for the remainder of the concentrations of these compounds, from the predicted value of the calibration curve, see calibration curves in appendix 2.
- Reproducibility will be monitored with the use of a range table or shewart plot as described in "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" USEPA, June 1972.

In general, the higher concentration standards should exhibit lower errors than the lower concentrations and the errors should be randomly distributed and the fit be approximately linear.

Range tables for this analysis are not completely statistically defined yet as the defining population for determination is not yet large enough. However; range tables have been provided with this method to illustrate the technique and to provide preliminary data for measuring the methods ability to provide reproducible data, see appendix 1. In the tables in appendix 1 you will also find the reproducibility data for the samples already analyzed by this method. Many of these points are above the upper control limits currently set. This could be partially due to the population being low or due to a more serious problem. During the time period allowed for approval of this method a greater population will be generated and other potential problems will be investigated by further analyzing the samples that we currently have.

As you will note from both the calibration curves and from the range tables benzene and naphthalene presented higher than expected errors for reproducibility. Unfortunately, time does not allow further statistical investigation of this until after this report is sent. It is believed that this situation can be corrected for by providing new calibrations for these compounds earlier than required for the other target compounds. This will be investigated during the approval period.

As standards are prepared by using the actual analytes verification of operation and calibration can be easily To help correct for errors due to injection all determined. analytes are divided by the internal standard, tetrachloroethene, and plotted as area ratios. If any samples have an internal standard area higher than that of the standards then the average internal standard area from the standards will be used for determining that samples area ratio. After initial verification at least three standards should be analyzed after every 10 samples at or near the quantitation limit, a mid range standard, and a standard at or near the highest concentration of the calibration curve. This is performed to insure that the calibration does not drift as a function of time. If these, "Calibration Check Compounds", as called by SW-846 Method 8270B section 7.4.4. page 15 differ less than or equal to 20 percent the initial calibration is assumed to be valid. If the criterion is not met for any one Calibration Check Compounds corrective action must be taken.

Since the standards are prepared in house and of good quality reagents we considered these to be the "true value" and matrix spikes will be used to determine the percent recovery or accuracy. Since the samples are sent to us pre-extracted, the spike is performed by injecting 30 ul of methylene chloride containing the target analytes into the extractant. It should be noted that this recovery only accounts for matrix interferences

in the methylene chloride extractant.

10 percent of the samples are analyzed in duplicate and 20 percent of the samples should be blanks.

## 8.1 Quantitation Limits:

Compound	Quantitation	Limit	ua/
1,1,1-trichloroethane	56		-31
benzene	76	•	
trichloroethene	61		
toluene	57	•	
ethylbenzene	54		
o-xylene	58		
decane	84	•	
1,3,5-trimethylbenzene	192		
undecane	206		
o-dichlorobenzene	207		
naphthalene	192		-

#### 9.0 GC/MS Analysis:

Once the standard or sample is in the autosampler vial a 7 ul injection of tetrachloroethene in methylene chloride is added to act as an internal standard to yield a concentration of 5636 ug/l. Samples that required dilution were diluted volumetrically. Direct injection of 2 ul of the methylene chloride phase is made into the injection port via a splitless injection on the GC/MS where separation and detection occur based on the conditions stated in sections 7.2 to 7.4. A sample chromatogram is given in appendix 3.

2 40.7 cm

#### 10.0 Data Interpretation:

# 10.1 Qualitative Analyses:

The qualitative identification of compounds determined by the method is based on retention time, and on comparison of the sample mass spectrum. In order to obtain the greatest sensitivity for these samples typically only one characteristic ion has been chosen for identification purposes. The relative retention time of the sample component should not exceed +/- 0.06 retention units.

#### 10.2 Quantitative Analyses:

When a compound has been identified, the quantitation of that compound will be based on the integrated abundance of that characteristic ion.

The calibration curve is expressed by the following equation

Concentration mg/l = Parameter 1 \* Area Ratio \*\* Parameter 2.

In order to determine the concentration of the sample the area

ratio, which is the area of the target analyte divided by the internal standard, is taken to the power of parameter 2, which should be very close to 1, and multiplied by parameter 1. This yields the concentration of the target analyte in the methylene chloride phase. This is the number that should be reported.

#### 11.0 Quality Control:

The requirements for this project are:

- 1. Before processing any samples, the analyst should demonstrate through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Since samples are being supplied with no reagent, trip, field etc. blanks it is impossible to assure that interferences from the glassware and reagents are under control.
- 2. The experience of the analyst is invaluable to the success of the method. Each day that the analysis is performed the daily calibration standards should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

3. A quality control reference sample concentrate is required containing each target analyte. The QC reference sample concentrate may be prepared from pure standard materials. This reference sample concentrate is then injected into methylene chloride and the recovery determined. This should be done at three concentrations one near the quantitation limit, one mid range, and at or near the highest concentration used for the calibration curve. For each analyte compare the standard deviation of the recovery in ug/l and the average recovery in ug/l with the corresponding acceptance criteria for precision and accuracy.

Compound	Ave. Recovery	Std. Dev.
1,1,1-trichloroethane benzene trichloroethene toluene ethylbenzene o-xylene decane 1,3,5-trimethylbenzene undecane dichlorobenzene naphthalene	100.15 100.32 100.74 100.26 100.37 101.14 101.86 100.98 101.65 102.40	3.34 3.60 7.25 5.11 7.76 8.62 11.03 7.35 10.25 13.00
_		*

4.3

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10 / . 10 .

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# Appendix 1 Range Tables

Volatile and Semivolatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique

Compound: 1,1,1-trichloroethane

Conc. Range mg/l	Sample f		UWL	UCL
0.06 - 0.07 - 3-0.07 - 0.15	1, 2, 3,	0.04, 0.02, 0.01 0.01, 0.03	0.07	0.09
0.15 - 0.45	, 4, 6, 9 <sub>3</sub>	0.03, 0.02, 0.02	0.07	0.09
0.45 - 0.89 ··· · · · · · · · · · · · · · · · · ·	41.001	المراجعة ومعالم المراجعة المراجعة ومعالم المراجعة ومعالم المراجعة ومعالم المراجعة ومعالم المراجعة ومعالم المرا المراجعة المراجعة ال	0.07 0.07	0.09 0.09
1.62 - 3.32	\$2.00 <u>0</u>		0.14	0.19
3.32 - 6.84	a7.00:	•	0.49	0.64
6.84 - 14.71 <sup>3</sup> 14.71 - 20.22	38.400	,	0.84	1.09
14.71 - 20.22	. TY 03.		<del></del>	
Compound: benzene	67 4 3 4 4 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	•		
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.08 - 0.10	47 SV1		0.08	0.10
0.10 - 0.20 0.20 - 0.61			0.08 0.08	0.10 0.10
0.61 - 1.20			0.28	0.36
1.20 - 2.19			0.31	0.40
2.19 - 4.48	v.		0.78	1.01
4.48 - 9.22			2.33	3.03
9.22 - 12.39		•	3.00	3.90
Compound: trichlore	oethene		· · · · · · · · · · · · · · · · · · ·	
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08	1	0:00	0.08	0.10
0.08 - 0.16		0.00	0.08	0.10
0.16 - 0.48	4, 5	0.02, 0.05	0.08	0.10
0.48 - 0.95	3	0.06	0.08	0.10
0.95 - 1.70 1.70 - 3.57		. *************************************	0.08 0.24	0.10
3.57 - 7.35	•		0.24	0.36
7.35 - 9.22			0.70	0.91
		•	•	-

Compound: toluene

	• '	The state of the s	•	***
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08			0.07	0.09
0.08 - 0.15	4 .	0.02	0.07	0.09
0.15 - 0.45	4 2, 3	0.00, 0.01	0.07	0.09
0.45 - 0.90		· ·	0.07	0.09
0.90 - 1.64	1	0.03	0.08	0.10
1.64 - 3.35	• ,	:	0.09	0.12
3.35 <b>-</b> 6.89		_	0.23	0.30
6.89 - 9.26	5	0.18	0.86	1.12
Compound: ethyl ber	nzene		,	
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.05 - 0.07		·	0.07	0.09
0.07 - 0.14			0.07	0.09
0.14 - 0.43	1, 2	0.03, 0.02	0.07	0.09
0.43 - 0.84		·	0.07	0.09
0.84 - 1.54			0.07	0.09
1.54 - 3.16	` <b>3</b>	0.16	0.16	0.21
3.16 - 6.50	4 .	0.07	0.29	0.37
6.50 - 8.73			0.90	1.17
Compound: o-xylene		and the second		-
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08		. — — — — — — — — — — — — — — — — — — —	0.07	0.09
0.08 - 0.15	3	0.02	0.07	0.09
	1, 2, 5	0.02, 0.02, 0.13	0.07	0.09
0.46 - 0.92	4, 7	0.08, 0.07	0.07	0.09
0.92 - 1.68			0.10	0.13
1.68 - 3.44			0.11	0.15
3.44 - 7.06	6	0.40	0.31	0.40
7.06 - 9.50			1.50	1.94
9.22 - 12.39			3.00	3.90
Compound: decane		A section		:
				_
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL.
0.03 - 0.20		·	0.11	0.14
0.20 - 0.22			0.11	0.14
0.22 - 0.67	3, 4, 5	0.24, 0.18, 0.05		0.14
0.67 - 1.33	1	0.20	0.11	0.14
1.33 - 2.43			0.12	0.16
2.43 - 4.98			0.29	0.37
4.98 - 10.24	2, 7	0.49, 1.41	0.37	0.48
10.24 - 13.76	6	1.75	1.55	2.02
•				

: bancamo	1.3	,5-trimethy	lbenzene
COMPAGNA			

•	* **		•	
Compound: 1,3,5-tri	•	•	w.,	
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08			0.07 0.07	0.09 0.09
0.08 - 0.15 0.15 - 0.46	<b>1</b>	0.04	0.07	0.09
0.46 - 0.91	1 2, 4	0.09, 0.15	0.07 0.10	0.09 0.13
0.91 - 1.67 1.67 - 3.42	5	0.90	0.14	0.13
	· 3	0.47	0.33	0.43
7.03 - 9.45			1.16	1:50
Compound: undecane		·		• .
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08			0.11	0.15
0.08 - 0.15			0.11	0.15
0.15 - 0.46		· • • • • • • • • • • • • • • • • • • •	0.11	0.15 0.15
0.46 - 0.91 0.91 - 1.67	4	0.09	0.14	0.18
1.67 - 3.42	3	0.26	0.12	0.16
3.42 - 7.03	2, 5	0.86, 1.01	0.32 0.52	0.83 0.68
7.03 - 9.45	-	0.007 1.01		
Compound: o-dichlor	cobenzene			
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.06 - 0.08		en e en	0.07	0.09
0.08 - 0.15	·		0.07 0.07	0.09 0.09
0.15 - 0.46 0.46 - 0.92	1.	0.29	0.31	0.40
0.92 - 1.68	2, 3	0.22, 0.21	0.17	0.22
1.68 - 3.43	4	0.39	0.76	0.99
3.43 - 7.06		•	1.29 1.74	1.68 2.27
7.06 - 9.49			1.74	2.21
Compound: naphthale	ene			
Conc. Range mg/l	Sample #	Sample Range	UWL	UCL
0.05 - 0.07		•	0.07	0.09
0.07 - 0.23		0.03.0.03	0.07 0.09	0.09
0.23 - 0.43 0.43 - 0.85	1, 2	0.03, 0.02	0.41	0.54
0.85 - 1.55			0.58	0.75
1.55 - 3.19	3 .	0.16	1.39	1.81
3.19 - 6.55	4	0.07	2.20 4.17	2.86 4.42
6.55 - 8.81	·	•		

# Appendix 2 Calibration Curves

Volatile and Semivolatile Organics by Gas chromatography/Mass Spectrometry (GC/MS):
Capillary Column Technique

7/06/95 111tca removing all about hard cet

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

#### BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .395917E+01 PARAMETER PAR(2) = .101459E+01

			•	
x	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.019	.056	.069	013	-18.695
.012	.056	.046	.011	23.694
.012	.056	.046	011	·- 23.490
.026	.092	.098	<del>`</del> ∴006	<del>-</del> 5.981×
.027	.092	.101	009	<del>-</del> 9.375
.025	.092	.093	002	-1.807
.027	.092	.101	009	-9.341
.022	.092	.080	.011	14.058
.022	.092	.082	.010	12.465
.033	.092	.126	034	-27.050
.031	.092	.117	026	-21.828
.059	.201	.225	024	-10.766
.057	.201	.218	017	-7.829
.055	.201	.207	006	-3.044
.057	.201	.216	016	-7.271
.185	.697	.715	018	-2.540
.183	.697	.707	010	-1.422
.185	.697	.715	018	-2.508
.191	.697	.740	043	-5.763
.187	.697	.723	026	-3.565
.285	1.079	1.107	028	-2.568
.280	1.079	1.088	009	809
.290	1.079	1.127	048	-4.236
.269	1.079	1.044	.035	3.317
.281	1.079	1.091	012	-1.067
.276	1.079	- 1.074 - ··	.005	458
.546	2.164	2.141	.023	1.054
.563	2.164	2.212	048	-2.188
.563	2.164	2.209	046	-2.065
.523	2.164	2.053	.111	5.400
1.158	4.485	4.594	110	-2.386
1.087	4.485	4.308	. 177	4.108
1.098	4.485	4.353	.132	3.034
1.125	4.485	4.462	.023	.515
·			•	

1.050	4.485	4.161	.324	7.777
1.096	4.485	4.346	.138	3.185
1.017	4.485	4.027	.458	11.375
2-203	9.187	8.823	.363	4.120
2.226	9.187	8.915	.272	3.047
2.202	9.187	8.818	.369	4.182
5.615	20.224	22.798	-2.573	-11.288

AVERAGE INDEPENDENT VARIABLE, XBAR = .613843

SUM OF THE SQUARES OF THE RESIDUALS = 7.38837

SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 40.9355

VARIANCE = .189445

STANDARD ERROR = .435253

STUDENT T VALUE = 2.0230 CORRESPONDING TO 39 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 39

### 95% CONFIDENCE INTERVALS

			• •	
X	Y	LOWER Y	PREDICTED Y	UPPER Y
.019	.056	091	.069	.229
.012	.056	115	.046	.206
.012	.056	115	.046	.206
.026	.092	062	.098	.257
.027	.092	058	.101	.261
.025	:092	066	.093	.253
.027	.092	058	.101	-261
022	.092	079	.080	.240
.022	.092	078	.082	.241
.033	092	033	.126	.285
.031	.092	042	.117	.277
.059	.201	.068	.225	.382
. 057	.201	.060	.218	.375
.055	.201	.049	-207	.365
. 057	.201	.059	.216	.374
.185	.697	.566	.715	.865
.183	.697	.557	.707	.857
.185	<b>.</b> 697 .	.565	.715	.865
.191	.697	.590	·740	.889
.187	.697	.573	.723	.872
.285	1.079	.963	1.107	1.252
.280	1.079	.943	1.088	1.233
.290	1.079	.982	1.127	1.271
.269	1.079	.899	1.044	1.190
.281	1.079	.946	1.091	1.235
.276	1.079	.929	1.074	1.219
.546	2.164	2.003	2.141	2.279
.563	2,164	2.074	2.212	2.350
.563	2.164	2,072	2.209	2.347

.523	2.164	1.915 4.438	2.053 4.594	· 4.751
1.158	4.485 4.485	4.156	4.308	4.460
1.098	4.485	4.200	4.353	4.505

• •	14 T	10 to 1	538.3	•
1.125	4.485	4.307	4.462	4.616
1.050	4.485	4:011	4.161	4.311
1.096	4.485	4.194	4.346	4.499
1.017	4.485	3.878	4.027	4.175
2.203	9.187	8.565	_ 8.823 <sub></sub>	9.081
2.226	9.187	8.654	1.0 1.0 1.0 1.0 1.0	9.176
2.202	9.187	8.560		9.076
5.615	20.224	22.096	22.798	23.500

7/06/95
Benzene complete ch. cet 1 & stal 11

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

#### BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .246008E+01 PARAMETER PAR(2) = .101516E+01

	•			•
X	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.026	.076	.061	.015	24.433
.030	.076	.069	.007	10.273
.029	.076	.068	<b>% 008</b>	11.768
.047	.076	.111	035	-31.651
.049	.124	.114	.009	8.246
.049	.124	.115	.009	7.395
.044	.124	.104	.020	19.367
.054	.124	.127	003	-2.151
.052	.124	.123	.001	.662
.061	.124	.145	021	-14.498
.072	.124	.171	047	-27.568
.061	.124_		019	-13.352
.077	.124	.183	059	<b>-32.233</b> .
.111	.271	.263	.008	2.924
.116	.271	.277	006	-2.148
.113	.271	.269	.002	.749
.123	.271	.293	022	<del>-</del> 7.533
.153	.271	.366	095	-26.031
.471	.940	1.145	205	-17.917
.370	. 940	.896	.044	4.934
.374	.940	<b>.</b> 906 ·	<b>V</b> 034	3.802
.395	.940	.959	019	-1.969
.449	.940	1.090	150	-13.763
.476	.940	1.157	217	-18.784
.520	1.455	1.267	.188	14.837
.552	1.455	1.345	.110	8.163
.562	1.455	1.370	.085	6.195
.558	1.455	1.359	.096	7.025
.597	1.455	1.458	003	187
:699	1.455	1.710	255	-14.889
1.430	2.918	3.536	618	-17.490
1.105	2.918	2.721	.196	7.213
1.179	2.918	2.908	009	.319
1.324	2.918	3.270	352	-10.768
		147		•

1.329	2.918	\		
2.137		3.285	367	-11.170
	6.048	5.318	.729	13.714
2.111	6.048	5.253		
2.160	6.048	_ , _ ,	.795	15.139
2.200		5.376	.671	12.489
	6.048	5.477	.571	10.417
3.608	6.048	9.050	-3.002	
2.268	6.048	5.650		-33.174
2.555	6.048		.398	7.042
5.762		6.376	328	-5.140
	12.389	14.557	-2.168	
4.332	12.389	10.897	<del>-</del>	-14.893
4.564	12.389		1.492	13.693
4.833		11.491	<b>.</b> 898 .	7.816
4.033	12.389	12.176	.212	1.744

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.09099
SUM OF THE SQUARES OF THE RESIDUALS = 19.9396
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 96.0403
VARIANCE = .453172
STANDARD: ERROR = .673180
STUDENT T VALUE = 1.9900 CORRESPONDING TO 44 DEGREES OF FREEDOM
ACTUAL DEGREES OF FREEDOM = 44

# 95% CONFIDENCE INTERVALS

X	Y	LOWER Y	2022	
.026	.076		PREDICTED Y	UPPER 3
.030	-076	184	.061	.307
.029	.076	176	.069	-314
.047		177	.068	.313
.049	.076	132	.111	.355
.049	-124	129	-114	.358
.044	.124	128	.115	.359
	.124	140	.104	.348
.054	, .124	117	.127	
052	.124	120	.123	.370
.061	.124	098	.145	.366
.072	.124	071	.171	-387
.061	.124	100		-413
.077	.124	059	.\.143	-385
.111	.271	_	7183	.424
.116	.271	.024	.263	.502
.113	.271	.038	.277	.515
.123	.271	.030	.269	.507
.153		.055	.293	.530
.471	.271	.130	.366	.601
.370	.940	.930	1.145	1.360
.374	.940	.675	-896	1.117
.395	.940	.685	•906	1.126
•	.940	.740	.959	1.178
.449	.940	.874	1.090	
.476	.940	.943	1.157	1.306
				1.372

.520	1.455	1.055	1.267	1.479
.552	1.455	1.134	1.345	1.556
<b>.</b> 562	1.455	1.160	1.370	1.580
.558	1.455	1.149	1.359	1.570

.597 1.455 1.249 1.458 1.666 **~** 699 1.455 1.505 1.710 1.914 1.430 2.918 3.333 3.536 3.739 1.105 2.918 2.524 2.721 2.919 1.179 2.918 2.711 2.908 3.106 1.324 · 2.918 3.070 3.270 3.470 1.329 2.918 3.084 3.285 3.485 2.137 6.048 5.075 5.318 5.562 2.111 6.048 5.011 5.253 5.494 2.160 6.048 5.131 5.376 5.622 2.200 6.048 5.228 5.477 5.726 3.608 6.048 8.653 9.050 9.447 2.268 6.048 5.395 5.650 5.905 2.555 6.048 6.094 6.376 6.657

13,888

10.412

10.976

11.628

14.557

.10.897

11.491

12.176

15.225

11.382

12.005

12.724

5.762

4.332

4.564

4.833

12.389

12.389

12.389

12.389

7/06/95
TCE complete who set 1

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1)\*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

### BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .437029E+01 PARAMETER PAR(2) = .100093E+01

<b>x</b> .	<b>Y</b> .	PREDICTED Y	RESIDUAL	PERCENT ERROR
.015	.061	.067	007	-10.077
.014	.061	.060	.000	.662
.023	.099	.102	003	-3.081
.023	.099	.099	\.000	436
.022	.099	.095	. 💃 003	3.339
.023	.099	.099	001	829
.023	.099	.102	004	-3.453
.019	-099	084	.015	17.328
.022	.099	.096	.003	2.916
.023	.099	.100	001	-1.132
.053	.216	.229	014	-5.964
.051	۶216	.220	005	-2.110
.048	.216	.210	.006	2.815
.048	216	.207	-008	3.983
.176	.749	.768	019	-2.502
.177''	.749	.771	022	-2.844
.175	.749	.765	016	-2.062
.178	.749	.776	027	-3.441

.266	1.160	1.161	001	097
.276	1.160	1.203	043	-3.589
.270	1.160	1.176	017	-1.437
.263	1.160	1.147	.012	. 1.078
.269	1.160	1.174	014	-1.213
.549	2.325	2.397	072	-3.002
.543	2.325	2.372	047	-1.961
.571	2.325	2.494	169	<del>-</del> 6.773
1.105	4.820	4.828	009	182
1.118	4.820	4.886	067	-1.369
1.078	4.820	4.711	.108	2.302
1.086	4.820	4.745	.075	1.573
1.047	4.820	4.574	.246	5.373
1.066	4.820	4.658	.161	3.466
2.192	9.873	9.587	.286	2.984
2.195	9.873	9.600	.272	2.838

AVERAGE INDEPENDENT VARIABLE, XBAR = .441288
SUM OF THE SQUARES OF THE RESIDUALS = .305247
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 11.4660
VARIANCE = .953896E-02
STANDARD ERROR = .976676E-01
STUDENT T VALUE = 2.0400 CORRESPONDING TO 32 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 32

#### 95% CONFIDENCE INTERVALS

x	· <b>Y</b> · ·	LOWER Y	PREDICTED	Y UPPER Y
.015	.061	.025	.067	.110
.014	.061	.018	.060	.103
.023	.099	.060	\.102	.144
.023	.099	.057	2,099	.141
.022	.099	.053	.095	.138
.023	.099	.057	.099	.142
.023	.099	.060	.102	.144
.019	.099	042	.084	.126
.022	.099	.054	.096	.138
.023	.099	.058	. 100	.142
.053	.216	.188	.229	.270
.051	.216	.179	.220	.262
.048	.216	.169	.210	.251
048	.216	.166	.207	.249
.176	.749	.731	.768	.806
.177	.749	.733	.771	.809
.175	.749	.727	.765	.802
.178	.749	.738	.776	.813
	•	151		•

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7/06/95
nap Complete 4/0 set 1
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INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1)\*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

#### BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .173233E+01 PARAMETER PAR(2) = .998549E+00

•		•		
x	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.031	.054	.055	001	-1.323
.032	.054	.055	001	<b>-1.976</b>
.031	.054	.054	• 000	407
.043	.088	.076	:012	16.302
.069	.088	.121	033	-27.117
.051	.088	.088	.000	080
.067	.088	.116	028	-23.979
.039	.088	.068	.020	29.476
.059	.088	.103	015	-14.686
.071	.088	.123	035	-28.424
.072	.088	.125	037	-29.617
.058	.088	.101	013	-12.927
.095	.192 -	166	.026	15.939
.092	.192	.160	.033	20.413
.101	.192	.175	.017	9.912
.135	.192	.235	042	-18.011
.140	.192	.244	051	-21.048
.314	.668	.545	.123	22.570
.385	.668	.667	.001	.118
.470	.668	.815	147	-17.993
.560	.668	.971	<b>−</b> ⁄-303	<del>-</del> 31.197
.529	.668	.917	249	-27.144
.468	1.034	.811	.223	27.456
470	1.034	.815	.219	26 <b>.</b> 928
.467	1.034	.810	.224	27.620
.546	1.034	.947	.087	9.163
$\times^{1.458}$	1.034	2.524	-1.490.	-59.029
.823	1.034	1.427	393	-27.531
1.237	2.074	2.142	068	-3.193
1.458	2.074	2.524	450	-17.835
1.715	2.074	2.969	895	<b>-</b> 30 <b>.157</b>
1.664	2.074	2.881	807	-28.024
2.086	4.299	. 3.610	-689	19.088
1.714	4.299	2.968	1.331	44.853

1.966	4.299	3.402	.896	26.347
2.020	4.299	3.495	.804	22.991
2.166	4.299	3.748	.550	14.680
2.895	4.299	5.007	709	-14.151
3.335	4.299	5.767	-1.468	-25.463
4.219	8.805	7.293	1.512	20.731
5.775	8.805	9.980	-1.174	-11.766
6.423	8.805	11.097	-2.291	-20.647

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.10359
SUM OF THE SQUARES OF THE RESIDUALS = 19.9640
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 94.9758
VARIANCE = .499101
STANDARD ERROR = .706471
STUDENT T VALUE = 2.0210 CORRESPONDING TO 40 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 40

## 95% CONFIDENCE INTERVALS

X	Y	LOWER Y	PREDICTED Y	UPPER Y
.031	.054	216	.055	.325
-032	.054	215	.055	.325
.031	.054	216	.054	.325
.043	.088	194	.076	.345
.069	.088	147	.121	.388
<b>.</b> 051 .	.088	181	.088	.357
.067	.088	152	.116	.383
.039	.088	202	.068	.338
.059	.088	165	.103	.371
-071	.088	144	.123	.390
.072	.088	142	.125	
.058	.088	167	.101	.392
.095	.192	099	.166	.369
.092	.192	106	.160	.431
.101	.192	090		.425
.135	.192	028	.175 .235	-440
-140	.192	018	.235	.497
.314	.668	,296	.545	.505
.385	.668	.423	.667	.794
.470	.668	.576	.815	.912
.560	.668	.737	.971	1.054 1.205
-529	.668	.681	.917	1.153
-468	1.034	.572	.811	1.051
-470	1.034	.576	.815	1.054
.467	1.034	.571	.810	1.050
. 546	1.034	.712	.947	1.182
.:458	1.034	2.298	2.524	2.750
.823	1.034	1.203	1.427	1.651
				1.001

1.237 1.458 1.715 1.664	2.074 2.074 2.074 2.074	1.921 2.298 2.731 2.646	2.142 2.524 2.969	2.363 2.750 3.207
•	2.074	2.040	2.881	3.116

			-	
2.086 1.714 1.966 2.020 2.166 2.895 3.335 4.219 5.775	4.299 4.299 4.299 4.299 4.299 4.299 8.805	3.346 2.730 3.148 3.237 3.479 4.665 5.373 6.787 9.261	3.610 2.968 3.402 3.495 3.748 5.007 5.767 7.293 9.980	3.873 3.205 3.656 3.753 4.018 5.350 6.161 7.800
6.423	8.805	10.287	9.980 11.097	10.699 11.90

# Appendix 3 Sample Chromatogram

Volatile and Semivolatile Organics by Gas chromatography/Mass Spectrometry (GC/MS):
Capillary Column Technique

201.0

114.2

185. C

CV3,g

3,38 . 8

144. 12

# **ATTACHMENT H-5**

# TOTAL PETROLEUM HYDROCARBONS DIESEL-RANGE ORGANICS

Total Petroleum Hydrocarbons

Diesel Range Organics

by Gas Chromatography/Mass Spectroscopy or Flame Ionization

Detection: Capillary Column Technique

This method includes sections directly copied from Wisconsin Department of Natural Resource Publication PUBL.-5W-141 Modified DRO Method for Determining Diesel Range Organics.

# 1.0 Scope and Application:

This method is designed to measure the concentration of diesel range organics in soil. This corresponds to a hydrocarbon range of  $C_{10}$  to  $C_{28}$  and a boiling point range between approximately 170°C and 430°C. Alterations to these core samples such as the use of surfactants may require changes to be made to this method.

The quantitation limit of this method is 2 mg/l per component of the Diesel Compound Standard in methylene chloride.

This method is based on a methylene chloride, pH adjusted water, soil extraction followed by gas chromatographic separation and either mass spectrometric or flame ionization detection. This method should be used by, or under the supervision of, analysts experienced in solvent extraction and the use of gas chromatographs. The analyst should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

This method is designed to measure mid-range petroleum products such as diesel or fuel oil. This method combined with Volatile and Semivolatile Organics by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique is believed to give an accurate representation of the contamination of the Hill AFB site.

# 2.0 Summary of Method:

This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as diesel, fuel oil #2, or kerosene. Samples analyzed are partially extracted prior to receipt and completed by sonication after receipt. The sample after sonication is centrifuged and the methylene chloride phase is removed for analysis by gas chromatography with either mass spectrometric or flame ionization detection. Quantification is based on response compared to a diesel component standard.

#### 3.0 Definitions:

#### Diesel Range Organics (DRO):

All chromatographic peaks eluting between n-decane  $(n-C_{10})$  and n-octacosane  $(n-C_{20})$ . Quantification is based on direct comparison of the area within this range to the total area of the 10 components in the Diesel Component Standard.

#### Diesel Component Standard:

## A ten component blend of typical diesel compounds:

Decane
Dodecane
tetradecane
hexadecane
octadecane
eicosane
docosane
tetracosane
hexacosane
octacosane

This standard mixture serves as a quantitation standard and a retention time window for diesel range organics.

#### Diesel Component Spike:

A reagent water or method blank sample spiked with the Diesel Component Standard and run with five percent of all samples as a quality control check. At a minimum one Diesel Component Spike must be run.

#### 4. Interferences:

Other organic compounds; including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the DRO results include these compounds. Spills of neat products should be quantified by specific analysis for the product in question.

Method interferences can be the result of contaminated glassware and reagents. Reagent blanks should be analyzed with each batch or for every 20 samples to demonstrate that the samples are free from method interferences. As the samples are being supplied partially extracted and all reagents are supplied from the site it is up to the people collecting the samples to provide these blanks. The laboratory will verify separately it reagents and glassware.

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by

analysis of a solvent blank to check for cross-contamination.

### 5.0 Apparatus and Materials:

Gas Chromatograph-Mass Spectrometer-Automatic Sampler-Data StationHewlett Packard 5890 with capillary injection Either a Hewlett Packard 5970 or 5972

Hewlett Packard ALS 7673

Hewlett Packard Chemstation or integrator J&W Scientific DB-1701, 30 meters, .25 mm

I.D., .26 um film thickness

Syringe- 10 ul

Balance- Analytical, 0.0001 g

Bottles- Glass with Teflon-lined screw caps or crimp

tops -

#### 6.0 Reagents:

Column-

Water- Millipore Corporation Milli-Q UV Plus Methylene Chloride- Aldrich Cat. # 32,399-3 99.9+% PRA Grade Organic Analytes- 97% pure or greater

Calibration Standards and Stocks-

prepared according to MTU-EEL Procedure entitled Standard Preparation Procedure 5/19/92 DLP or prepared from a stock purchased for Aldrich Chemical Co.

MS Tuning Standard- Perfluorotributylamine

# 7.0 Sample Collection, Preservation, and Handling:

This is to be performed by others, method unknown. Once samples are received at MTU-EEL they are stored at 4°C for not more than 47 days.

#### 8.0 Procedure:

#### 8.1 Sample Preparation:

In attempt to insure complete extraction from the soil/water matrix into the methylene chloride all samples are sonicated, at room temperature, in our sonicator at 21 kilocycles per second for 15 minutes. After sonication the samples are centrifuged at 2300 revolutions per minutes for 15 minutes to separate the phases. After phase separation a 1.0 ml aliquot is removed from the vial and put into an HP autosampler vial with a Teflon lined septum and crimp cap.

# 8.2 Operating Conditions for the HP 5890 GC:

Injection port- 250°c
Oven Temp Initial Time- 2 minutes
Oven Temp Initial Value- 35°c
Oven Temp Program Rate- 10°c
Oven Temp Final Time- 6 minutes

3.50 (2.50 )

Oven Temp Final Value- 275°C
Transfer Line Temp- 280°C
Column Head Pressure- 8.5 psi
Approximate Column Flow Rate- .9 ml/minute
Septum Purge Flow Rate- 8.0 ml/minute
Split Vent Flow Rate- 31.3 ml/minute
Splitless Time- .5 minutes

# 8.3 Operating Conditions of the HP ALS 7673:

Sample WashSample PumpsSample VolumeViscosity DelaySolvent A (methylene chloride) WashSolvent B (methylene chloride) Wash3
Solvent B (methylene chloride) Wash3

# 8.4 Operation Conditions of the HP Mass Selective Detector and Plame Ionization Detector:

Electron Multiplier Voltage (Relative to Autotune) - 0
Scan Range - 50 to 850
Solvent Delay - 3.4 minutes
Average Pressure - 2.6 X 10<sup>-5</sup>
Flame Ionization Detector Temperature - 250°C

#### 9.0 Calibration:

A minimum of a five point calibration curve must first be analyzed to verify proper operation of the GC/MS or FID. After fitting the standards all analytes must meet acceptance criteria. This criteria includes:

- 1. percent relative standard deviations of an expected less than 30 percent, which is used to determine if the GC/MS or GC/FID is exhibiting deterioration of response (as determined in SW-846 Method 8270B section 7.3.4.1 page 13) based on the DRO Standard
- deviation of any single concentration point greater than 30%, this 30 percent error is only an educated guess based on limited data, from the predicted value of the calibration curve. A preliminary calibration curve is included in appendix 1.
- Reproducibility will be monitored with the use of a range table or shewart plot as described in "Handbook for Analytical Quality Control in Water and Wastewater Laboratories" USEPA 1972:

Range tables are not yet defined for this method. The best that can be said at this time is that the highest error in reproducibility yet seen for this method so far is 23 percent.

In general, the higher concentration standards should exhibit lower errors than the lower concentrations and the errors should be randomly distributed and the fit be approximately linear.

As standards are prepared by using the actual analytes verification of operation and calibration can be easily determined. After initial verification at least three standards should be analyzed after every 10 samples at or near the quantitation limit, a mid range standard, and a standard at or near the highest concentration for the calibration curve. This is performed to insure that the calibration does not drift as a function of time. If these "Calibration Check Compound", as called by SW-846 Method 8270B section 7.4.4. page 15, differ less than or equal to 20 percent the initial calibration is assumed to be valid. If the criterion is not met for the Calibration Check Compound corrective action must be taken.

Since the standards are prepared in house and of good quality reagents, stock purchased from Aldrich Chemical Co., we considered these to be the "true value" and matrix spikes will be used to determine the percent recovery or accuracy. Since the samples are sent to us pre-extracted the spike is performed by injecting 30 ul of methylene chloride containing the DRO Standard into the extractant. It should be noted that this recovery only accounts for matrix interferences in the methylene chloride extractant.

10 percent of the samples are analyzed in duplicate and 15 percent of the samples should be blanks.

#### 9.1 Quantitation Limits:

The quantification limit for this method should be 2 mg/l per component of the DRO Standard.

### 10.0 GC/MS Analysis:

Samples that require dilution will be diluted volumetrically. Direct injection of 2 ul of the methylene chloride phase is made into the injection port via a splitless injection on the GC/MS or FID where separation and detection occur based on the conditions stated in sections 8.2 to 8.4.

# 11.0 Data Interpretation:

# 11.1 Qualitative Analyses:

The qualitative identification of compounds determined by the method is based on retention time. All chromatographic peaks eluting between n-decane and n-octacosane should be added together and reported.

Quantitation is based on direct comparison of the area within this range to the total area of the ten components in the Diesel

Component Standard. The retention time window is defined as beginning approximately 0.1 minutes before the retention time of n-decane and ending 0.1 minutes after the retention time of n-cotacosane in the calibration run.

If there are significant peaks outside the chromatographic window, this fact must be reported.

If there is a rise in the baseline but no peaks in the chromatogram report no detect (ND), but note the raise in the baseline in the comments section of the report. If there are peaks you must quantitate and report the results as DRO. However, if the peaks represent a small percentage of the total DRO peak area as compared with the area associated with the raise in the baseline then you may flag the data and qualify it in the comments section of the report.

# 11.2 Quantitative Analyses:

When qualitative analyses parameter are met the quantitation of that sample will be based on the integrated abundance of that scan in MS or the raw area in the case of the FID..

The calibration curve is expressed by the following equation

Concentration mg/l = Parameter 1 \* Total Area \*\* Parameter 2.

In order to determine the concentration of the sample the total area is taken to the power of parameter 2, which should be very close to 1, and multiplied by parameter 1. This yields the concentration of the DRO in the methylene chloride phase. This is the number that should be reported.

# 12.0 Quality Control:

The requirements for this project are:

- 1. Before processing any samples, the analyst should demonstrate through the analysis of a method blank, that interferences from the analytical system, glassware, and reagents are under control. Since samples are being supplied with no reagent, trip, field etc. blanks it is impossible to assure that interferences from the glassware and reagents are under control.
- 2. The experience of the analyst is invaluable to the success of the method. Each day that the analysis is performed the daily calibration standards should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal?; Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard

chromatogram can indicate whether the column is still good, the injector is leaking, the injector septum needs replacing, etc. If any changes are made to the system (e.g. column changed), recalibration of the system must take place.

3. A quality control reference sample concentrate is required containing each target analyte. The QC reference sample concentrate may be prepared from pure standard materials or purchased. This reference sample concentrate is then injected into methylene chloride and the recovery determined. This should be done at three concentration one near the quantitation limit, one mid range, and one at one at or near the highest concentration used for the calibration curve. The average recovery should be approximately 100% and the standard deviation should be approximately 8%, we are currently determining these recoveries.

# Appendix 1 Calibration Curve

Total Petroleum Hydrocarbons Diesel Range Organics by Gas Chromatographic/Mass Spectrometry of Flame Ionization Detection: Capillary Column Technique

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: ENDENT VARIABLE, X, IS: area DEPENDENT VARIABLE, Y, IS: conc. mg/L

EQUATION OF THE FORM: Y=PAR(1)\*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS
BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .577528E-09 PARAMETER PAR(2) = .114951E+01

X ******** ******* ******* *******	Y 1.974 5.118 10.304 20.911 10.304 20.911 48.396	PREDICTED Y 1.749 4.285 13.094 26.930 12.081 22.760	RESIDUAL .226 .833 -2.790 -6.019 -1.777 -1.850	PERCENT ERROR 12.907 19.434 -21.307 -22.350 -14.7098.126
	48.396	39.408	8.988	`´22.807

AVERAGE INDEPENDENT VARIABLE, XBAR = .125057E+10
SUM OF THE SQUARES OF THE RESIDUALS = 132.111
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = .462382E+19
VARIANCE = 26.4222
STANDARD ERROR = 5.14025
STUDENT T VALUE = 2.5710 CORRESPONDING TO 5 DEGREES OF FREEDOM
ACTUAL DEGREES OF FREEDOM = 5

### 95% CONFIDENCE INTERVALS

X ******** ******* ******* *******	Y 1.974 5.118 10.304 20.911 10.304 20.911	LOWER Y -6.527 -3.008 7.902 20.497 6.757 17.195	PREDICTED Y 1.749 4.285 13.094 26.930 12.081 22.760	UPPER Y 10.024 11.578 18.286 33.362 17.404 28.326
******	48.396	29.409	22.760 - 39.408	28.326 49.407

DRO 7/28/95

DEPENDENT VARIABLE, X, IS: area DEPENDENT VARIABLE, Y, IS: conc. mg/L

# Appendix 2 Sample Chromatogram

Total Petroleum Hydrocarbons Diesel Range Organics by Gas Chromatographic/Mass Spectromerty of Flame Ionization Detection: Capillary Column Technique

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.266	1.160	1.125	1.161	1.196
.276	1.160	1.167	1.203	1.238
.270	1.160	1.141	1.176	1,212
.263	1.160	1.111	1.147	1.183
.269	1.160	1.138	1.174	1.209
.549	2.325	2.362	2.397	2.432
.543	2.325	2.337	2.372	2.406
.571		2.459	2.494	2.529
1.105	4.820	4.777	4.828	4.880
1.118	4.820	4.834	4.886	4.939
1.078	4.820	4.660	4.711	4.762
1.086	4.820	4.694	4.745	4.796
1.047	4.820	4.524	4.574	4.623
1.066	4.820	4.608	4.658	4.708
2.192	9.873	9.478	9.587	9.695
2.195	9.873	9.492	9.600 测数	9.709

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Toluene complete who let I t std 7 from set 7

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

#### BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .199447E+01 PARAMETER PAR(2) = .101000E+01

			•	•
<b>X</b> .	Υ .	PREDICTED Y	RESIDUAL	PERCENT ERROR
.03Ò	.057	.057	.000	841
.027	.057	.053	.004	7.975
.025	.057	.049	.008	16.061
.024	.057	.046	.011	23.911
.050	.093	.097	004	-4.595
. 053	.093	.102	010	-9.331
.052	.093	.101	008	-8.365
.049	.093	.095	002	-2.371
.051	.093	.099	007	-7.014
.045	.093	.088	.005	5.364
.050	.093	.097	005	-4.997
.050	.093~	.097	004	-4.422
.051	.093	.098	005	<del>-</del> 5.377
.112	.202	.218	016	-7.122
.111	.202	.217 `	015	-6.878
.108	.202	.211	009	-4.166
.100	.202	.194	.008	4.215
.107	.202	.209	006	-3.062
.372	.703	.734	032	-4.307
.373	.703	.736	<b>-</b> √. 033	-4.493
.363	.703	.717	014	-2.013
.373	.703	.737	035	-4.690
.364	.703	.719	017	-2.312
.562	1.088	1.114	026	-2.376
.565	1.088	1,120	032	-2.891
.569	1.088	1.129	041	-3.648
.558	1.088	1.106	018	-1.629
.568	1.088	1.127	039	-3.482
1.118	2.182	2.232	050	<del>-</del> 2.251
1.105	2.182	2.206	025	-1.111
1.117	2.182	2.230	048	-2.167
1.104	, 2.182	2.205	023	-1.048
2.241	4.522	4.505	017	.375
2.241	4.522	4.506	.015	.342

2.230	4.522	4.484	.038	.845
2.234	4.522	4.492	.030	.674
2.153	4.522	4.327	.195	4.510
2.185	4.522	4.391	.131	2.974
2.142	4.522	4.304	.217	5.052
4.511	9.263	9.134	.129	1.409
4.333	9.263	8.770	.493	5.620
4.135	9.263	8.366	.897	10.724

AVERAGE INDEPENDENT VARIABLE, XBAR = .919342
SUM OF THE SQUARES OF THE RESIDUALS = 1.18586
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 62.0272
VARIANCE = .296465E-01
STANDARD ERROR = .172181
STUDENT T VALUE = 2.0210 CORRESPONDING TO 40 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 40

.027	24 19 16 13 63 68 67 61
.027       .057      014       .053       .1         .025       .057      018       .049       .1         .024       .057      021       .046       .1         .050       .093       .031       .097       .1         .053       .093       .036       .102       .1         .052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	16 13 63 68 67 61 65
.025       .057      018       .049       .1         .024       .057      021       .046       .1         .050       .093       .031       .097       .1         .053       .093       .036       .102       .1         .052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	13 63 68 67 61 65
.024       .057      021       .046       .1         .050       .093       .031       .097       .1         .053       .093       .036       .102       .1         .052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	63 68 67 61 65
.050       .093       .031       .097       .1         .053       .093       .036       .102       .1         .052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	68 67 61 65
.053       .093       .036       .102       .1         .052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	68 67 61 65
.052       .093       .035       .101       .1         .049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	67 61 65
.049       .093       .029       .095       .1         .051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	65
.051       .093       .033       .099       .1         .045       .093       .022       .088       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	
.045 .093 .022 .088 .1 .050 .093 .031 .097 .1 .050 .093 .031 .097 .1 .051 .093 .032 .098 .1 .112 .202 .153 .218 .2 .111 .202 .153 .217 .2 .108 .202 .147 .211 .2 .100 .202 .129 .194 .2	
.050       .093       .031       .097       .1         .050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	54
.050       .093       .031       .097       .1         .051       .093       .032       .098       .1         .112       .202       .153       .218       .2         .111       .202       .153       .217       .2         .108       .202       .147       .211       .2         .100       .202       .129       .194       .2	63
.051     .093     .032     .098     .1       .112     .202     .153     .218     .2       .111     .202     .153     .217     .2       .108     .202     .147     .211     .2       .100     .202     .129     .194     .2	63
.112     .202     .153     .218     .2       .111     .202     .153     .217     .2       .108     .202     .147     .211     .2       .100     .202     .129     .194     .2	64
.111 .202 .153 .217 .2 .108 .202 .147 .211 .2 .100 .202 .129 .194 .2	82
.108 .202 .147 .211 .2 .100 .202 .129 .194 .2	82
.100 .202 .129 .194 .2	76
	59
	73
.372 .703 .676 .734 .7	93
·.373 .703 .677 .736 .7	95
.363 .703 .658 .717 .7	76
.373 .703 .679 .737 .7	96
.364 .703 .660 .719 .7	79
.562 1.088 1.058 1.114 1.1	
.565 1.088 1.064 1.120 1.1	76
.569 1.088 1.073 1.129 1.1	85
.558 1.088 1.050 1.106 1.1	62
.568 1.088 1.071 1.127 1.1	83

1.118	2.	182	2.177	2.232	2.286
1.105		182	2.152	2.206	2.260
1.117		182	2.175	2.230	2.284
1.104	_	182	2.175	2.230	2.259

	• • • • • • • • • • • • • • • • • • • •			
2.241	4.522	4.426	4.505	4.584
2.241	4.522	4.427	4.506	4.586
2.230	4.522	4.405	4.484	4.563
2.234	4.522	4.413	4.492	4.571
2.153	4.522	4.250	4.327 march	4.403
2.185	4.522	4.314	4.391	4.469
2.142	4.522	4.228	4.304	4.381
4.511	9.263	8.967	9.134	9.302
4333	9.263	8.610	8.770	8.930
4.135	9.263	8.214		8.518
		•		Sales and the sales are

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Ethylbenzene complet 4/0 set 1

INDEPENDENT VARIABLE, Y, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .154434E+01 PARAMETER PAR(2) = .993846E+00

•	1 2 2 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1				
<b>X</b> ,	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR	
.033	.054	.052	.001		
.036	.054	.057	004	2.711 <del>-</del> 6.651	
.029	.054	.046	.008		
.029	.054	.046	.007	17.102	
.057	.087	.090		15.518	
.061	.087	.095	003	-3.110	
.057	.087	.090	008 003	-8.388	
.057	.087	.090	~.002	-2.840	
.060	.087	.094	007	-2.637	
.051	.087	.081	.006	-7.200	
.056	.087	.088	001	7.865	
.062	.087	.097	009	<b>943</b>	
.055	.087	.087		<del>-</del> 9.750	
.122	.191	.192	.000	.483	
.129	.191	.202	001	451	
.123	.191	.192	012	<del>-</del> 5.773	
.123	.191		001	588	
.114	.191	.192	001	588	
.125	.191	.179	.012	6.510	
.426		.195	005	-2.31 <u>4</u>	
.431	.662	.661	, .001	.186	
.443	.662	.669	₹-007	978	
.460	.662	.688	026	-3.755	
.444	.662	.714	<b>~.</b> 052	<del>-</del> 7.268	
.669	.662 1.025	.690	027	-3.949	
.682	1.025	1.036	011	-1.050	
.670		1.055	030	-2.838	
.672	1.025	1.038	012	-1.193	
.694	1.025	1.040	015	-1.463	
:691	1.025	1.074	049	<del>-4</del> .591	
1.344	1.025	1.069	044	-4.097	
1.387	2.056	2.071	015	747	
1.403	2.056	2.137	081	-3.799	
1.372	2.056	2.162	106	-4.896	
1.3/2	2.056	2.115	060	-2.817	
		171			

2.701	4.261	4.146	.115	2.778
2.778	4.261	4.263	001	032
2.795	4.261	4.289	028	648
2.722	4.261	4.178	.084	1.999
2.674	4.261	4.105	.157	3.819
2.610	4.261	4.008	.254	6.332
2.675	4.261	4.106	.155	3.776
5.619	8.729	8.586	.143	1.668
5.574	8.729	8.517	.212	2.486
5.242	8.729	· 8.013	.716	8.942

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.10359
SUM OF THE SQUARES OF THE RESIDUALS = .744904
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 99.3303
VARIANCE = .177358E-01
STANDARD ERROR = .133176
STUDENT T VALUE = 1.9980 CORRESPONDING TO 42 DEGREES OF FREEDOM
ACTUAL DEGREES OF FREEDOM = 42

X	Y	LOWER Y	PREDICTED Y	UPPER Y
.033	.054	.003	.052	.101
.036	.054	.008	.057	.107
.029	.054	004	.046	.095
.029	.054	003	.046	.096
.057	.087	.041	.090	.139
.061	.087	.046	.095	.144
.057	.087	.041	.090	.139
.057	.087	.041	.090	.138
.060	.087	.045	.094	.143
.051	.087	.032	081	.130
.056	.087	.039	088	.137
.062	.087	.048	.097	.145
.055	.087	.038	.087	.136
.122	.191	.144	.`192	.239
.129	.191	.155	-202	.250
.123	.191	.144	.192	.240
.123	.191	.144	.192	.240
.114	.191	.131	.179	.227
.125	.191	.147	.195	.243
.426	.662	.617	.661	.705
.431-	.662	.625	.669	.713
.443	. 662	.644	.688	.732
.460	.662	.671	.714	.758
.444	.662	.646	.690	.733
.669	1.025	.994	1.036	1.078
.682	1.025	1.013	1.055	1.097

.670-	1.025	.996	1.038	1.079
.672	1.025	.999	1.040	1.082
. 694	1.025	1.033	1.074	1.116
.691	1.025	1.027	1.069	1.111

1.344	2.056	2.031	2.071	2.112
1.387	2.056	2.096	2.137	2.178
1.403	2.056	2.121	2.162	2.203
1.372	2.056	2.075	2.115	2.156
2.701	4.261	4.088	4.146	4.205
2.778	4.261	4.203	4.263	4.323
2.795	4.261	4.229	4.289	4.350
2.722	4.261	4.119	4.178	4.237
2.674	4.261	4.047	4.105	4.163
2.610	4.261	3.951	4.008	4.064
2.675	4.261	4.048	4.106	4.164
5.619	8.729	8.459	8.586	8.713
5.574	8.729	8.392	8.517	8.643
5.242	8.729	7.895	8.013	8.130

		•		
2.654	4.634	4.720	086	-1.823
2.641	4.634	4.696	062	
. 2 612			002	-1.321
2.612	- 4.634	4.645	011	239
2.567	4.634	4.566	.068	
2.503			.008	1.480
	4.634	4.452	.181	4.069
2.398	4 (24			4.005
	4.634	4.267	.367	8.600
5.336	9.492		•	0.000
	3.436	9.446	-046 .	.487
4.978	9.492	8.815		
		0.073	. 677	7.675
4.759	9.492	8:430	3 844	- · · <del>-</del>
<del></del>		. 01430	1.061	12.591

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.05343

SUM OF THE SQUARES OF THE RESIDUALS = 1.80709

SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 84.5420

VARIANCE = .440753E-01

STANDARD ERROR = .209941

STUDENT T VALUE = 2.0020 CORRESPONDING TO 41 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 41

		:		
X	. <b>Y</b>	LOWER Y	PREDICTED Y	UPPER Y
-035	.058	015	.064	.143
.035	• 058	016	.063	.142
.027	.058	029	.050	.130
.025	.058	033	.046	.126
.050	.095	.013	.092	.170
.057	•095 ·	.025	.103	
.055	.095	.022	.100	.182
.055	.095	.021	.100	.179
.057	.095	.026	.105	.179
.051	.095	.014		.183
.056	.095	.023	.093	.172
-056	.095	.022	.102	.181
.047	.095	.007	.101	-180
-119	.207	.138	-086	.165
.127	.207	.154	.215 \.231	.292
.120	.207	.140	217	.308
.108	.207	·119	.197	294
.110	.207	.123	200	-274
.416	.720	.679		. 277
.420	.720	.684	.749 .755	.819
.407	.720	.661	.732	.825
.417	.720	.679		.802
.407	.720	.663	.749	.820
652	1.115	1.104	.733	.804
.650	1.115	1.100	1.170	1.237
.651	1.115	1.101	1.166	1.233
.648	1.115	1.095	1.168	1.235
	<b></b>	± • U3 3	1.162	1.229

7/06/95 Xylene Complete to such

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1)\*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

## BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .178909E+01 PARAMETER PAR(2) = .993643E+00

	2			
X	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
• 035 <sup>-</sup>	.058	.064	006	-8.885
.035	.058	.063	005	<del>-</del> 7.653
.027	.058	.050	,008	16.240
.025	.058	.046	012	25.824
. 050°	- 095	.092	.003	3.524
.057	- 095	.103	009	-8.269
.055	.095	.100	006	-5.487
.055	.095	.100	005	-5.058
.057	.095	.105	010	-9.458
.051	.095	.093	.002	1.951
.056	.095	.102	007	-7.097
.056	.095	.101	006	-6.265
.047	.095 -	.086	.009	10.570
.119	. 207	.215	008	-3.626
.127	.207	.231	024	-10.242
.120	.207	.217	009	-4.371
.108	.207	.197	.011	5.391
.110	.207	.200	.007	3.587
.416	.720	.749	029	-3.849
<b>.</b> 420 .	.720	.755	035	-4.578
.407	.720	.732	-,011	-1.561
.417	.720	.749	- 029	-3.909
.407	.720	.733	013	-1.777
.652	1.115	1.170	056	-4.761
.650	1.115	1.166	052	-4.427
.651	1.115	1.168	053	-4.552
.648	1.115	1.162	047	-4.078
.636	1.115	1.142	027	-2.363
<b>.</b> 626	1.115	1.123	008	751
1.315	2.235	2.349	113	-4.826
1.253 ;	2.235	2.239	003	153
1.273	2.235	2.275	039	-1.720
1.238	2.235	2.212	.023	1.048
2.649	4.634	4.710	~.076	-1.620

		•	★ A THAT A THA	
.636	1.115	1.075	1.142	1.209
.626	1.115	1.056	1.123	1.190
.1.315	2.235	2.284	2.349	2.414
1.253	2.235	2.174	2.239	2.304

1.273	2.235	2.210	2.275	2.339
1.238	2.235	2.148	2.212	2.277
2.649	4.634	4.613	4.710	4.807
2.654	4.634	4.622	4.720	4.817
2.641	4.634	4.599	4.696	4.792
2.612	4.634	4.549	4.645	4.741
2.567	4.634	4.472	4.566	4.660
2.503	4.634	4.360	4.452	4.545
2.398	4.634	4.178	4.267	4.355
5.336	9.492	9.240	9.446	9.651
4.978	9.492	8.624	8.815	9.005
4.759	9.492	8.249	8.430	8.611

7/06/95 Decane complete W/o Set 1

INDEPENDENT VARIABLE, Y, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE PORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT \*ERRORS

## BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .270308E+01 PARAMETER PAR(2) = .101705E+01

:			• (	
X	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.037	.084	.096	011	-11.599
.031	.084	.078	.006	7.700
.047	.084	.122	₹.037	-30.530
.051	.138	.132	.006	4.515
.060	.138	.154	017	-10.903
.054	.138	.140	002	-1.734
.053	.138	.137	-000	.329
.057	.138	.147	009	-6.291
.049	.138	.126	.012	9.517
.074	.138	.191	053	-27.941
.051	.138	.132	.006	4.308
.043	.138	.109	.028	26.009
.112	.301	.292	.009	.2.974
.121	-301	.316	016	-4.964
.108	.301	.280	020	7.254
.105	.301	.272	.028	10.381
.113	.301	.294	.007	2.232
.388	1.044	1.031	.013	1.230
.395	1.044	1.052	008	<b>723</b>
.390	1.044	1.038	.006	.545
-400	1.044	1.064	020	-1.859
.388	1.044	1.032	.012	1.204
.558	1.616	1.494	.122	8.196
.614	1.616	1.646	~.030	-1.847
.618	1.616	1.657	040	-2.445
.600	1.616	1.607	7009	.550
.618	1.616	1.657	041	-2.445
.587	1.616	1.573	.043	2.762
1.258	3.241	3.412	171	-5.025
1.235	3.241	3.351	110	-3.289
1.232	3.241	3.342	101	-3.026
1.168	3.241	3.167	.074	2.345
2.441	6.718	6.700	.017	.257
2.388 -	6.718	6.551	.167	2.542
•				2.72

0 401	4	c .		-1.761
2.491	6.718	6.838	120	
2.531	6.718	6.950	232	-3.343
2.462	6.718	6.759	041	604
2.463	6.718	6.760	042	624
2.300	6.718	6.305	.413	6.548
5.129	13.761	14.255	494	-3.465
5.062	13.761	14.068	307	2.181
4.580	13.761	12.706	1.055	8.304

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.03482

SUM OF THE SQUARES OF THE RESIDUALS = 1.80842

SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 78.6294

VARIANCE = .452104E-01

STANDARD ERROR = .212627

STUDENT T VALUE = 2.0210 CORRESPONDING TO .40 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 40

X	Y	LOWER Y	PREDICTED	Y UPPER Y
.037	.084	.014	.096	.178
.031	.084	004	.078	
.047	.084	.040	.122	.203
.051	.138	.050	.132	.213
.060	.138	.073	.154	.236
.054	.138	.058	.140	.221
.053	.138		.137	.219
.057	.138	.065	.147	.228
.049	.138	.044	.126	.207
.074	.138	.110	.191	.272
.051	.138	.050	.132	.213
.043	.138	.027	.109	.191
.112	.301	.212	.292	.372
.121	.301	.237	.316	.396
.108	.301	.200	\.280	.360
.105	.301	.192	. 272	353
.113	.301	.214	.294	.374
.388	1.044	<b>1.958</b>	1.031	1.105
.395	1.044	.979	1.052	1.125
.390	1.044	.965	. 1.038	1.112
.400	1.044	.991	1.064	1.137
.388	1.044	.958	1.032	1.105
.558	1.616	1.423	1.494	1.564
.614	1.616	1.577	1.646	1.716
.618	1.616	1.587	1.657	1.726
.600	1.616	1.538	1.607	1.677
.618	1.616	1.587	1.657	1.726
.587	1.616	1.503	1.573	1.642

1.258	3.241	3.345	3.412	3.480
1.235	3.241	3.284	3.351	3.418
1.232	3.241	3.275	3.342	3.409
1.168	3.241	3.100	3.167	3.233

2.441	6.718	6.605	6.700	6.796
2.388	6.718	6.458	6.551	6.644
2.491	6.718	6.741	6.838	6.935
2.531	6.718	6.852	6.950	7.048
2.462	6.718	6.663	6.759 - "	6.854
2.463	6.718	6.664	6.760	6.856
2.300	6.718	6.215	6.305	6.395
5.129	13.761	14.046	14.255	14.464
5.062	13.761	13.862	14.068	14.274
4.580	13.761	12.522	12.706	12.890

112.3-

878.1-870.41

731. T. -

200. -

300.4 020. 107.5

10.0

7/06/95
1,3,5-trimethylbenzene complete 4/2 set (

INDEPENDENT VARIABLE, X, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1)\*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ERRORS

## BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .183211E+01 PARAMETER PAR(2) = .100135E+01

		•		
X	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.035	.058	.063	005	-7.929
.031	.058	.057	.002	2.778
.025	.058	.046	.012	24.979
.049	.058	.090	031	-35.150
.057	.094	.104	009	-8.838
.066	.094	.120	026	-21.323
.057	.094	.104	009	-8.999
-057	.094	.103	009	-8.516
.054	.094	.098	~.004	-3.926
.046	.094	.084	.010	12.353
.063	.094	.114	020	-17.163
.049	.094	.090	.005	5.479
.046	.094	.083	.011	13.337
.124	.206	.227	020	-8.847
.125	.206	.228	022	-9.432
.106	.206	.195	.012	
.094	.206	.172	.034	6.153
.100	.206	. 183	.023	19.906
.399	.717	.729	->p12	12.611 -1.654
.402	.717	.735	018	-2.390
.396	.717	.724	007	932
.387	.717	.709	.008	1.167
.411	.717	.753	035	-4.695
.637	1.110	1.166	056	-4.767
.650	1.110	1.190	079	-6.675
.631	1.110	1.155	045	-3.875
.622	1.110	1.139	029	-2.513
.614	1.110	1.125	014	-1.274
-595	1.110	1.090	.020	1.831
1:308	2.226	2.398	172	<b>-7.153</b>
1.218	2.226	2.232	005	228
1.195	2.226	2.190	.036	1.653
1.217	2.226	2.231	005	203
2.637	4.615	4.838	223	-4.606

2.570	4.615	4.714	099	-2.105
2.513	4.615	4.611	.004	.091
2.556	4.615	4.689	074	-1.587
2.486	4.615	4.560	.055	1.196
2.411	4.615	4.423	.192	4.335
2.362	4.615	4.332	.283	6.529
5.141	9.453	9.439	.014	.153
4.877	9.453	8.955	.499	5.569
4.677	9.453	8.587	.866	10.090

AVERAGE INDEPENDENT VARIABLE, XBAR = 1.02551
SUM OF THE SQUARES OF THE RESIDUALS = 1.23519
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 80.1908
VARIANCE = .301266E-01
STANDARD ERROR = .173570
STUDENT T VALUE = 2.0020 CORRESPONDING TO 41 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 41

X	Y	LOWER Y	PREDICTED Y	UPPER Y
.035	.058	002	.063	.129
.031	.058	009	.057	.122
.025	.058	019	.046	.112
.049	.058	.024	.090	.155
.057	.094	.039	.104	.169
.066	.094	.055	.120	.185
.057	.094	.039	.104	.169
.057	.094	.038	.103	.168
.054	.094	.033	.098	.163
.046	.094	.019	.084	.149
.063	.094	.049	.114	.179
.049	.094	.024	.090	.155
.046	.094	.018	.083	.149
.124	.206	.163	.227	.290
.125	.206	.165	228	.291
.106	.206	.131	.195	.258
.094	.206	1.108	.172	.236
.100	.206	.119	.183	.247
.399	.717	.671	.729	.788
.402	.717	.677	.735	.793
.396	.717	.666	.724	.782
.387	.717	.651	.709	.768
.411	.717	.695	.753	.811
: 637	1.110	1.111	1.166	1.221
.650	1.110	1.135	1.190	1.245
.631	1.110	1.100	1.155	1.210
.622	1.110	1.084	1.139	1.210

.614	1.110	1.069	1.125	1.180
.595	1.110	1.035	1.090	1.146
1.308	2.226	2.344	2.398	2.452
1.218	2.226	2.178	2.232	2.285

102.	: 7		
121			
	9 ti	• • • • • • • • • • • • • • • • • • •	
		* :•	".
1.195	2.226 :::	2.137	2.190 2 2.244
1.217	2.226	2.177	2.231 2.285
2.637	4.615	4.756	4.838 4.920
2.570	4.615	4.634	4.714 4.794
2.513	4.615	4.532	4.611 4.689
2.556	4.615	4.610	4.689 4.769
2.486	4.615	4.483	4.560 4.638
2.411	4.615	4.348	4.423 4.499
2.362	4.615	4.258	4.332 4.406
5.141	9.453	9.271	9.439 9.607
4.877	9.453	8.796	8.955 9.113
4.677	9.453	8.436	8.587 8.738

AVERAGE INDEPENDENT VARIABLE, XBAR = .564256

SUM OF THE SQUARES OF THE RESIDUALS = .735634

SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 19.3472

VARIANCE = .229886E-01

STANDARD ERROR = .151620

STUDENT T VALUE = 2.0400 CORRESPONDING TO 32 DEGREES OF FREEDOM

ACTUAL DEGREES OF FREEDOM = 32

X	Y	LOWER Y	PREDICTED Y	UPPER Y
.029	.058	.030	.095	.161
.012	.058	025	.041	.106
.034	.094	.049	, .114	.179
.040	.094	.067	<b>`.132</b>	.196
.038	.094	.061	.126	.190
.041	.094	.071	.135	.200
.033	.094	.044	.109	.174
.023	.094	.010	.076	.141
.026	.094	.023	.088	.153
.031	.094	.036	.101	.166
.070	.206	.168	.231	.295
.069	.206	.167	.230	.294
.062	.206	.142	.206	.270
.058	.206	.129	.193	.257
.227	.717	.691	.749	.807
.225	.717	.682	.740	.798
.217	.717	.657	.715	.774
.204	.717	.613	.672	.731
.351	1.110	1.099	1.154	1.210
.345	1.110	1.079	1.134	1.189
.351	1.110	1.098	\1.153	1.208
.296	1.110	.917	. 973	1.029
.345	1.110	1.078	1.133	1.188
-698	2.226	2.230	2.284	2.338
.674	2.226	2.154	2.208	2.262
.702	2.226	2.245	2.299	2.353
1.511	4.615	4.843	4.928	5.013
1.291	4.615	4.140	4.213	4.287
1.388	4.615	4.449	4.528	4.606
1.404	4.615	4.502	4.581	4.660
1.243	4.615	3.986	4.058	4.129
1.375	4.615	4.407	4.485	4.563
2.950	9.453	9.413	9.589	9.765
2.822	9.453	9.007	9.175	9.342

7/06/95 Dichlorobenzene

complete 46 set 1

INDEPENDENT VARIABLE, Y, IS: area ratio DEPENDENT VARIABLE, Y, IS: concentration MG/L

EQUATION OF THE FORM: Y=PAR(1) \*X\*\*PAR(2)

PARAMETERS FOUND BY MINIMIZING THE SUM OF SQUARES OF THE PERCENT ER

A STEEL

## BEST FIT PARAMETER VALUES

PARAMETER PAR(1) = .264168E+01 PARAMETER PAR(2) = .939865E+00

X	Y	PREDICTED Y	RESIDUAL	PERCENT ERROR
.019	.058	.064	006	-9.374
.011	.058	.039	.020	_
.031	.095	.102		50.404
.039	.095	•	007	-6.908
.036		.125	030	-24.110
	.095	.117	022	~18.802
.036	.095	.116	\021	-18.378
.041	.095	.133	2.038	-28.587
.024	.095	·.079	. 015	
.046	.095	.146		19.484
.026	.095	.085	051	-35.040
.062	.207		.010	11.633
.081		.194	013	6.756
	.207	.250	042	-16.998
.066	.207	.206	.001	.540
.063	.207	.195	.012	6.275
<b>, 257</b>	.720	.736	016	_
∵293	.720	.833		-2.134
.311	.720	•	113	-13.546
.326		.882	161	-18.308
	, .720	,922	202	-21.912

-397	1.115	1.108	.007	500
.401	1.115			•593
.423		1.118	004	328
— <del>-</del>	1.115	1.177	<b>~.</b> 062	-5.274
.425	1.115	1.183	068	-5.756
. 475	1.115	1.311	197	-14.987
•985	· 2.235	2.604	369	-14.159
•921	. 2.235	2.445	210	_
.970	2.235			-8.582
1.640	7,7 7	2.567	33%	-12.912
	4.634	. 4.205	-428	10.188
1.291	4.634	3.358	1.275	37.978
1.618	4.634	4.152	.482	11.602
1.670	4.634	4.278		
1.727	· -		•356	8.321
	4.634	4.414	.219	4.969
1.956	4.634	4.964	<b>~.3</b> 30	-6.648
3.415	9.492	8.380		
3.779	9.492		1.111.	13.261
	3.436	9.216		2.994

AVERAGE INDEPENDENT VARIABLE, XBAR = .701818
SUM OF THE SQUARES OF THE RESIDUALS = 4.06275
SUM OF THE SQUARES OF THE DEVIATIONS FROM XBAR = 29.8166
VARIANCE = .126961
STANDARD ERROR = .356316
STUDENT T VALUE = 2.0400 CORRESPONDING TO 32 DEGREES OF FREEDOM ACTUAL DEGREES OF FREEDOM = 32

X	Y	LOWER Y	PREDICTED Y	UPPER Y
.019	.058	090	.064	
.011	• 058 °	116		.219
.031	.095	051	.039	. 194
.039	.095		.102	.255
.036		028	.125	.278
	.095	036	.117	.270
.036	- 095	037	.116	.269
.041	.095	020	. 133	.285
.024	.095	<b></b> 075	•079	
.046	.095	006	· · · · ·	.233
.026	.095	~.069	.146	.298
.062	.207		.085	.239
.081		-043	-194	.345
<del>-</del>	-207	.100	.250	.399
.066	.207	-056	.206	.357
.063	.207	.044	.195	
.257	.720	.598	.736	.346
.293	.720	.697		.674
.311	.720		.833	• •969
.326		.747	.882	1.017
	.720	.788	•922	1.057

#### APPENDIX B

## FLUORESCEIN TRACER TEST RESULTS

APPENDIX B
Fluorescein Tracer Test Results

11/-	II Leve	19-Ap					20-Ar	x 20-A	or 20-Ap	r 20-Ap	r 20-Apı	20-Ap
ID			16:38	18:32	20:30	22:35	0:30	2:30	7:27	10:30	14:30	18:40
	(Colo		2.13	4.03	6.00	8.08	10.00	12.00	16.95	20.00	24.00	28.17
	blue	-5.60E+00							1 -1.00E-0	-2.00E-01		
	red			0 5.89E+0	1 1.07E+02	2	1.16E+0			1 4.20E+01	1.80E+01	
	white	1.12E+01	5.62E-0	1 7.50E+0	1 7.84E+01	1.14E+02				4.50E+01	3.27E+01	
	yellow	7.60E+00	1.01E+0	2 1.69E+0	1 6.34E+01	5.35E+01				3.95E+01	2.07E+01	
	1 black	-3.80E+00										7.18E-01
	blue	5.60E-01		1 3.44E+0 0 2.83E+0	1 4.26E+01			11	3.80E+01		1.97E+01	
	red	1.06E+01	9.07F+0	0 1.13E+0		4.34E+00		1.10E+0				
	white	8.15E+01	6.71E+0					1 4.82E+0		2.19E+01		
	yellow	-2.70E+00				2./1E+01	1.75E+0	1.56E+0		1.54E+01	1.35E+01	7.51E+00
3	1 black			1 1.70E+0 0 1.68E+0	6.79E+01	8.17E+01	7.14E+0			1.40E+01	6.41E+00	
	blue	-3.70E+00					1.22E+02		4.12E+01	1.52E+01	2.60E+00	-1.50E+00
	red	6.50E+00		1.55E+0	2.17E+02	1.43E+02			1.90E+00	-1.60E+00	-3.70E+00	-4.00E+00
	white	2.58E+01			1.14E+02			9.94E+01	4.41E+01	2.86E+01	1.94F+01	2.27E+01
	yellow	2.27E+02			1.08E+02	1.12E+02		6.22E+01	3.84E+01	1.72E+01	1.88F+01	1 94F±01
11	2 black	2.275+02	2.31E+02	2 1.59E+02	5.61E+01			4.41E+00	-1.30E+00	-2.60E+00	-3.90E+00	-4.50F+00
	blue	<del></del>	-4.80E+00	1.82E+01				1.27E+02	6.08E+01	3.24E+01	1.15E+01	2.32E+00
	red	<del> </del>	-2.40E+UC	-1.20E+00		3.17E+00	3.82E+00	1.45E+00	4.27E+00	1.44E+01	4.99E+01	5.85E+01
	white	<del> </del>		1.28E+01		2.35E+01		3.16E+01	2.52E+01	2.59E+01	3.10E+01	
	yellow	<del> </del>	4.30E+01	4.90E+01	7.79E+01	7.26E+01		6.71E+01	6.75E+01	6.31E+01	4.87F+01	
20	black	<del> </del>	3.73E+01	7.24E+01	1.05E+02	1.01E+02		5.62E+01	2.87E+01	1.93E+01	1.06F±01	6.46E+00
	blue	<del> </del>	-4.60E+00	-2.40E+00	-1.40E+00		3.62E+01	1.04E+02	1.25E+02	1.01E+02	7.03E+01	4.47E+01
	red	<del> </del>			-1.20E+00	1.00E+01	6.97E+00	1.72E+01	2.99E+01	2.38E+01	1.43E+01	1.41E+01
	white	<del> </del>	1.15E-01									
	vellow	<del> </del>		1.04E+02			7.25E+01	2.69E+01	5.10E-02	-2.70E+00	-3.90E+00	-4.20F±00
- 00	black		7.29E+01	1.60E+02			7.08E+01	3.54E+01	4.60E+00	-2.00E-01	-3.30E+00	-4 20F+00
-32		<del> </del>	-2.60E+00	-3.00E+00	2.20E+01	6.55E+00					6.78E+01	V.LUL TOO
	blue			<u> </u>								
	red	<del>  </del>		1.85E+01	2.41E+01	3.02E+01	4.74E+01	2.58E+01	1.01E+01	1.57E+01	1.90E+01	2 32F+01
	white	<del>  </del>	3.25E+00		3.33E+01	7.00E+U1	6.08E+01	6.11E+01	6.90E+01	4.10E+01	1.63E+01	1.38F+01
12	yellow black	<del>  </del>	1.99E+02	1.40E+02	7.68E+01	4.75E+01	2.69E+01	1.76E+01	5.67E+00	2.20E+00	-1.30E+00	-1.50E+00
13	blue	<del>  </del>										1.002100
	red	<u> </u>		0.005.00								
	white	<del>  </del>		6.80E+00	-1.60E+00	-2.00E+00	-2.00E+00	-1.20E+00	8.20E-01	1.34E+01	3.56E+01	7.69E+01
	yellow			2.36E+01	1.11E+01	2.06E+01	1.36E+01	1.84E+01	2.30E+01	3.74E+01		5.50E+01
23	black			1.14E+02	1.57E+02	1.39E+02	1.03E+02	6.32E+01	3.13E+01	2.07E+01		9.09E+00
	blue			-3.20E+00	-2.40E+00	-1.70E+00	4.48E+00			7.98E+01		5.39E+01
_	red			-3.20E+00	-2.90E+00	-2.30E+00	-2.00E-01	4.52E+00		3.11E+01		5.45E+01
	white			1.49E+00	1.84E-01	8.00E-02	1.38E+00	4.08E+00	1.04E+01	1.71E+01		2.28E+01
_	yellow			3.64E+00	7.87E+01	1.38E+02	1.39E+02	8.91E+01		2.82E+01		1.08E+01
	black .			6.48E+01	1.02E+02	1.05E+02	9.59E+01	7.36E+01	3.82E+01	2.32E+01		9.24E+00
	blue			-3.80E+00	-3.40E+00	-3.50E+00	3.60E+00	-3.40E+00	9.76E-01			8.90E+01
_				-1.50E+00	8.72E+00	3.14E+01	5.15E+01		7.16E+01		4.38E+01	2.67E+01
	red			1.61E+01	1.15E+01	1.19E+01	1.11E+01	1.49E+01	1.38E+011	1.35E+01	1.18F+01	1.46E+01
_	white			4./UE+00	1.49E+01	4.91E+01	8.03E+01	8.97E+01	9.80E+01	8.13E+01	4.40F±01	1.97E+01
	yellow			1.92E+02	1.27E+02	7.99E+01	4.81E+01	2.85E+01		2.39F+00 ·	1.80E+00 -	2 60F+00
_	black			-1.00E-01	7.49E-01		7.96E-01	4.32E+00	6.67E-01	4.57E-01		7.28E+01
$\overline{}$	blue											
	red			7.44E+00	8.23E+00	1.08E+01	1.27E+01	9.36E+00	6.54E+00	5.80E+00	1.83E+00	3 28F+00
	white			I.OULTOU;	~~.UUL+UU	1.42E+UU1	5.ZUE #011	1.151-4021	1 49E+091	6.95E+01	1.83E+00 2.56E+01	1 30E+01
	yellow				U.7 ULTU 11	1.1064021		74/H4331	4 705-1011		4 COE . O.4 L .	
	black			-2.30E+00	-1.60E+00  -	·1.70E+00  -	1.50F±00 .	1 30F 100	5.46E .00	4 045 . 04	0.005 04	
_	blue			U. / ULTUU	~4.500=+001 •	'J.JUE+UU! •	7.54UF+(X)1	5 19F-10BL	3 51F1041	4 E3E - 04	4 005 04	4 455
	red			2.104700	- 1.00E+001 •	'2.UUC+UUI -	ZINJE ARNII .	.1 701-1001	-9 20E - 00 i	4 01E 00	4 000 04	
	white			J.OULTOO	J.Z 1L+U11	1.00C+UZ1	7.401-11171	1 641-1021	2715.041	COAE.AAI.	0.000	
	/ellow			7.202.7001	1.046.4011	3.0/E+U11	1 234-7111	1 311-1021	1 025 . 021	4745.041	4	
34 T	olack			1.70	2.001-011	1.1/2+01/	1 ンントムまり	1 (745-104)	1 175.011	4 005 04 .	4	
14	olue		!:									
_	200			1 54E+00	1.76F+00							
r	ed										O'OTETINI A	
r V	vhite			-3.90E+00								
y y	vhite vellow			-3.90E+00 - 3.46E+01	5.40E+01	6.48E+01	3.17F+01	9.73F±01	9.26E+01			
7 V 51	vhite vellow 51			3.90E+00 3.46E+01 -1.30E+00	5.40E+01 1.72E+00	6.48E+01 8	3.17E+01	9.73E+01	9.26E+01 7.59E+01	8.24E+01 4 4.78E+01	1.98E+01 2 1.93E+01 8	.91E+01 .38E+00
y y	vhite vellow			3.90E+00 - 3.46E+01 -1.30E+00 1.73E+01	5.40E+01 1.72E+00 5.26E+01	6.48E+01 8 8.90E+00 1	3.17E+01 1.86E+01	9.73E+01 2.28E+01	9.26E+01 7.59E+01 4.04E+01	8.24E+01 4 4.78E+01		2.91E+01 3.38E+00 2.22E+01

## Fluorescein Tracer Test Results (Continued)

		21-Ap	r 21-Ap	21-Ap	22-Ap	r 22-Ap	r 22-Ap
We	l Leve	2:1					
	(Colo	r) 35.75	44.00	52.25	57.67	66.50	72.00
$\perp$	11 black	-4.90E+0		-5.50E+00	-5.40E+0	0	-4.90E+00
<u></u>	blue	3.73E+0		-1.70E+00	-1.80E+0	0	-2.80E+00
$\vdash$	red	1.90E+0			2.71E+0		1.03E+00
<b> </b>	white	5.31E+00			-5.00E-0	1	-1.30E+00
<u> </u>	yellow	1.57E-0			-3.10E+00	ol	-4.30E+00
1-	21 black	6.20E+00		<del></del>			
⊢-	blue	4.19E+01				)	
<b> </b>	red	3.04E+01			<del></del>		3.05E+00
<b>├</b> ─	white	1.44E+01					-1.00E+00
<u> </u>	yellow	9.62E-01					-3.80E+00
با	11 black		-4.50E+00			<u> </u>	-3.10E+00
├	blue		-5.30E+00				<u> </u>
<u> </u>	red	1.71E+01		8.60E+00			<u> </u>
	white	2.40E+01		1.46E+01			<u> </u>
<del>                                     </del>	yellow		-4.80E+00				L
<del>  '</del>	2 black		-3.50E+00	-2.90E+00			
$\vdash$	blue	6.72E+01		2.23E+01	1.50E+01		1.20E+01
├─	red	0.055.04	1.49E+01	7.34E+00	2.56E+01		6.87E+00
<del> </del>	white yellow	3.25E+01	1.71E+01 -1.20E+00	1.25E+01	9.13E+00		-1.00E-01
<del>                                     </del>	2 black	1.81E+01			-3.20E+00		-4.90E+00
	blue	2.41E+01	2.74E+01	0.00E+00	-9.00E-01		-1.10E+00
	red	2.712701	2.740+01	2.51E+01	1.88E+01		4.56E+00
	white	-4 40F+00	-5.50E+00	-5 20E+00		<del>                                     </del>	4.405.00
_	yellow	-4.80E+00	-5.50E+00	-5.20E+00	-5.70E+00		-4.40E+00
3:	2 black	1.002.100	O20LT00	-3.00LT00	-3.70E+00		-5.70E+00 1.04E+01
	blue	i					1.04E+01
	red.	2.93E+01	1.77E+01	2.42E+01	1.83E+01		1.85E+01
	white	6.29E+00	-1.70E+00				-2.10E+00
	yellow			-5.20E+00	-5.30E+00		-5.30E+00
13	black						
	blue						-
	red	5.80E+01	3.38E+01	2.15E+01	8.71E+00	1.04E+01	1.08E+01
	white	5.67E+01	3.89E+01	2.41E+01	1.27E+01	1.04E+01	1.18E+01
	yellow	2.67E+00	-5.00E-01	1.04E+00		3.50E+00	1.12E+00
23	black	3.29E+01	1.40E+01	8.33E+00	3.52E+00	1.65E+00	1.49E+00
	blue	3.47E+01	1.39E+01	8.22E+00	6.41E+00	6.44E+00	1.13E+01
	red	2.56E+01		1.91E+01	1.88E+01	1.41E+01	9.59E+00
-1.	white		-2.20E+00		-3.60E+00	5.75E-01	2.27E+00
	yellow	7.37E+00	3.00E+00	1.59E+00	3.85E+00	7.46E+00	6.42E-01
33	black	3.14E+01	1.62E+00		-2.90E+00		-3.80E+00
	blue	1.14E+01	3.10E+00	2.45E+00	5.78E-01	2.87E+00	1.93E+00
	red	2.83E+01	3.33E+01	2.34E+01	2.17E+01	2.61E+01	2.07E+01
	white	7.65E+00	2.00E+00		-9.00E-01	2.45E+00	0.00E+00
14	yellow black	-3.60E+00			4.90E+00		-3.60E+00
- 14	blue	8.62E+01	2.91E+01	1.07E+01	6.90E+00	3.87E+00	2.31E+00
	red	8.37E+00	1.49E+01	0.005.04			6.84E+00
	white	8.42E+00		2.62E+01 3.80E+00	3.83E+01		3.76E+01
	yellow	3.74E+00	0.00E+00	1.80E+00	1.43E+00	3.41E+00	1.67E+00
	black	5.04E+01	2.14E+01	8.52E+00	6 51E - 00		
	blue	3.16E+01			6.35E+00	3.23E+00 2.67E+00	1.04E+00
	red	3.24E+01			1.71E+01		4.30E-01 6.74E+00
	white		4.90E+00 -	5.20E+00 -	5.30E+00	4.90F-00	-5.60E+00
	vellow	-2.40E+00	4.30E+00 -	5.00E+00 -			-5.30E+00
	black	7.02E+01		3.62E+01	1.47E+01		3.94E+00
	blue	2.77E+01		1.67E+01	1.71E+01		4.84E+00
	red	8.28E+00			3.05E+01		2.00E+01
]	white	2.40E+01			5.29E+00		2.10E+00
	yeliow						4.90E+00
51	51	2.04E+01			1.09E+01		6.47E+00
52	52	1.13E+01	6.83E+00	8.83E+00	4.35E+00		-1.00E-01
53	53	2.76E+01	1.90E+01		1.04E+01		4.84E+00

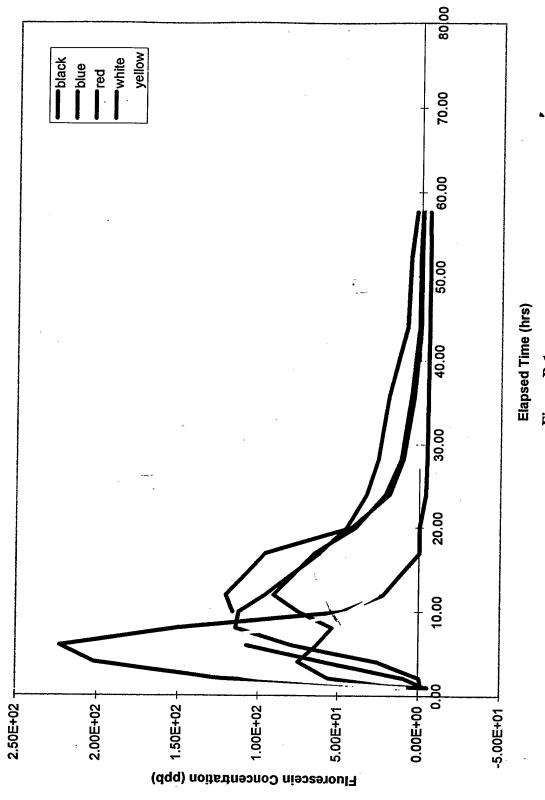


Figure B-1.

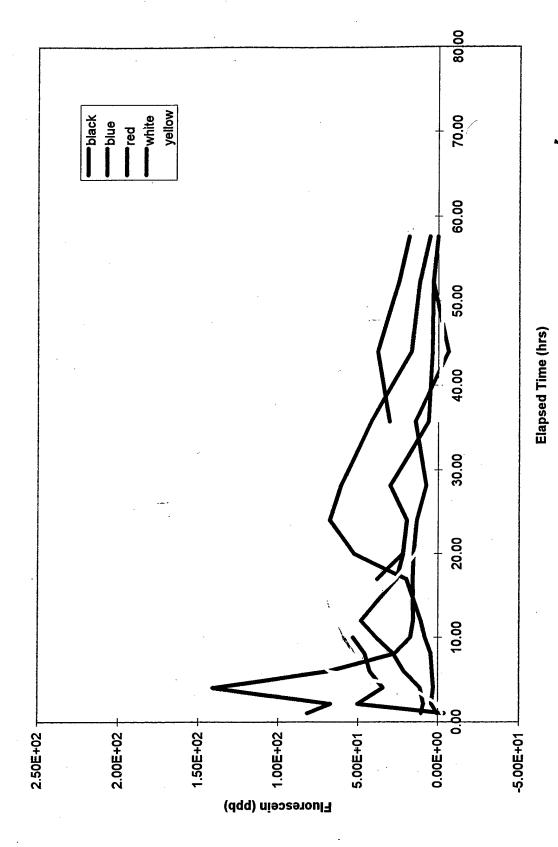
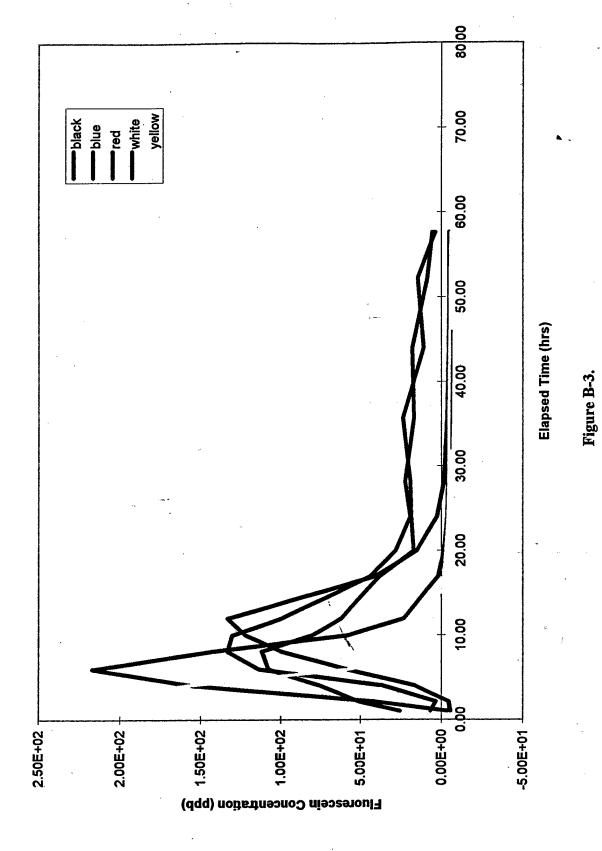


Figure B-2.



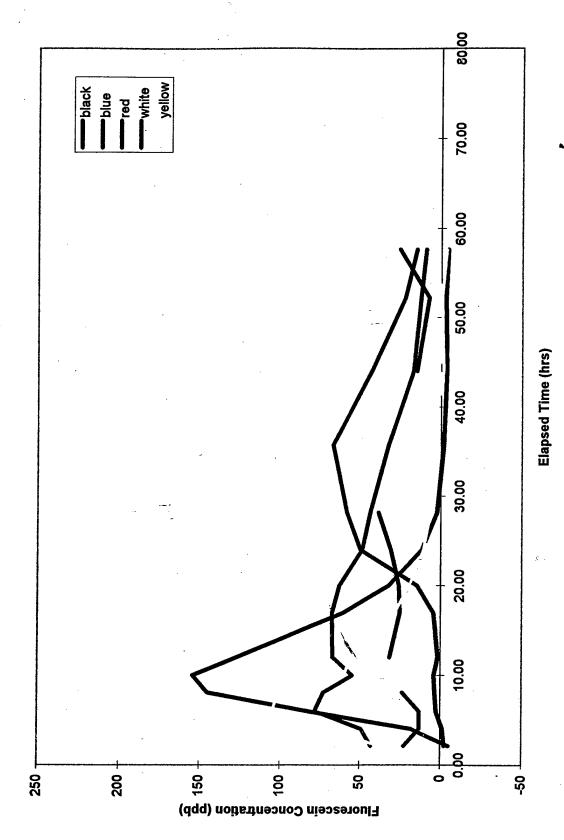


Figure B-4.

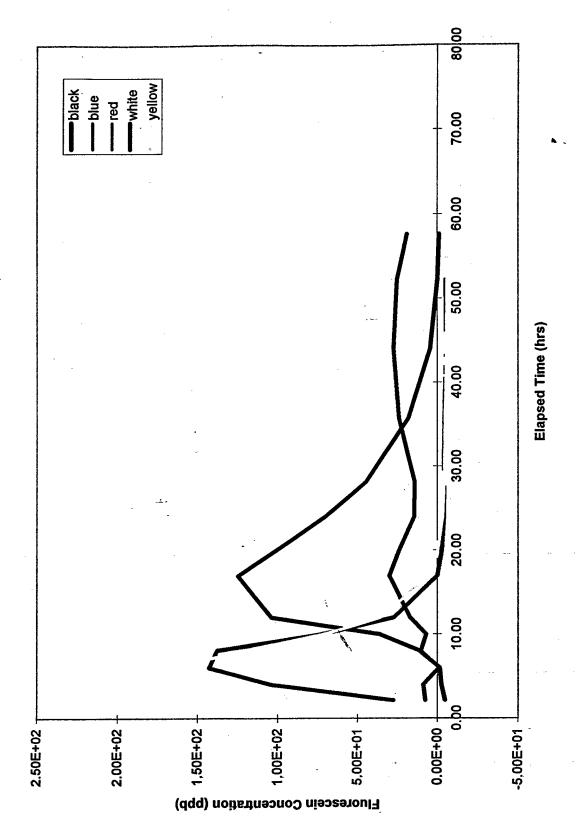


Figure B-5.

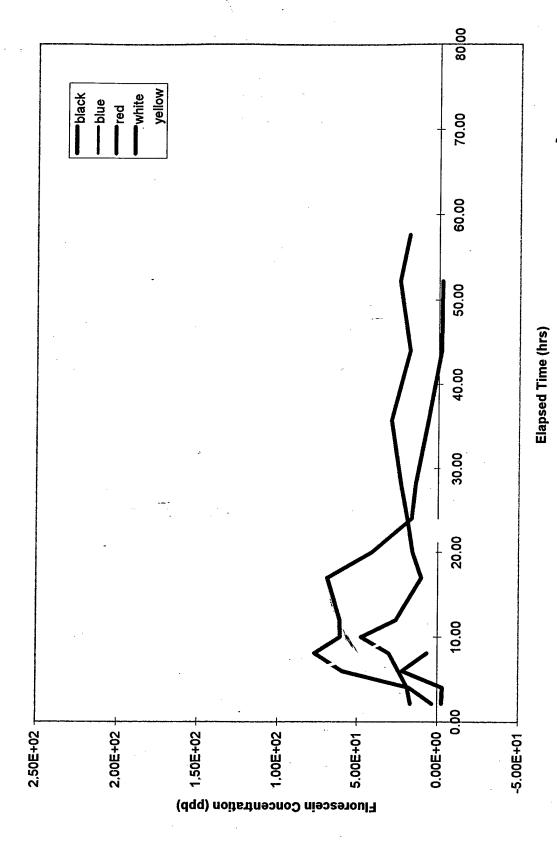


Figure B-6.

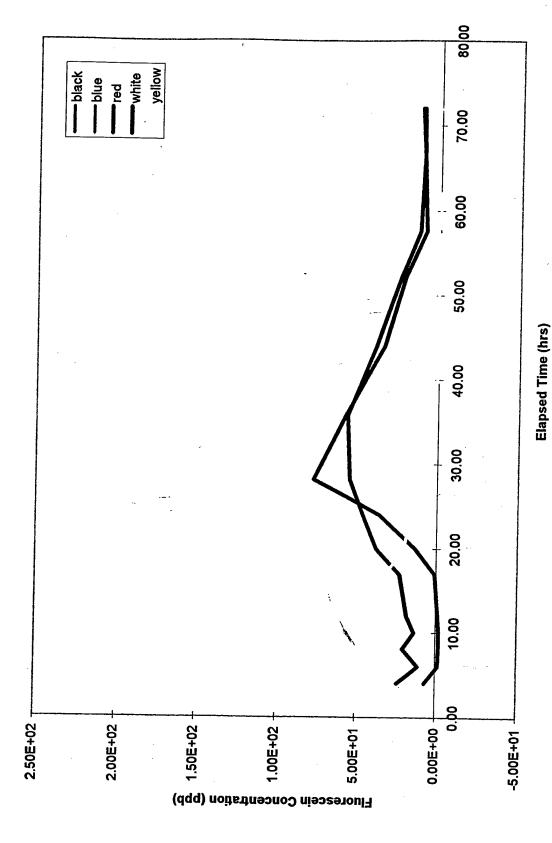


Figure B-7.

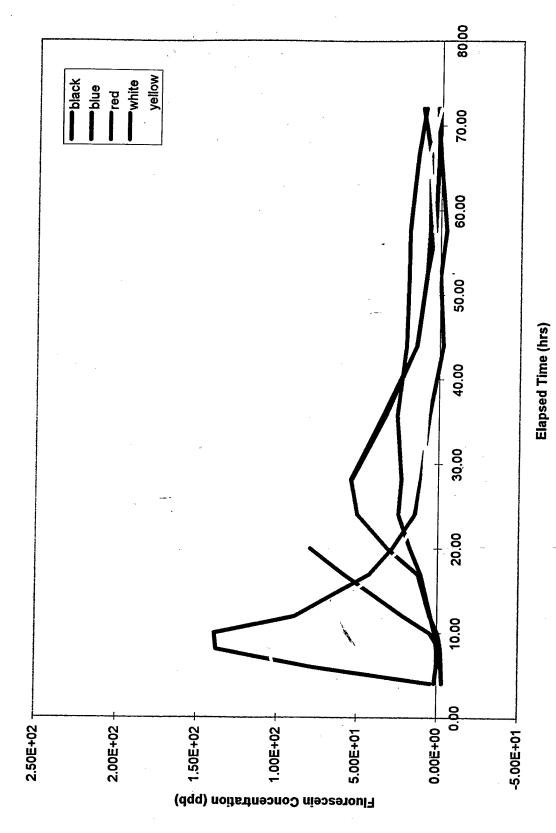


Figure B-8.

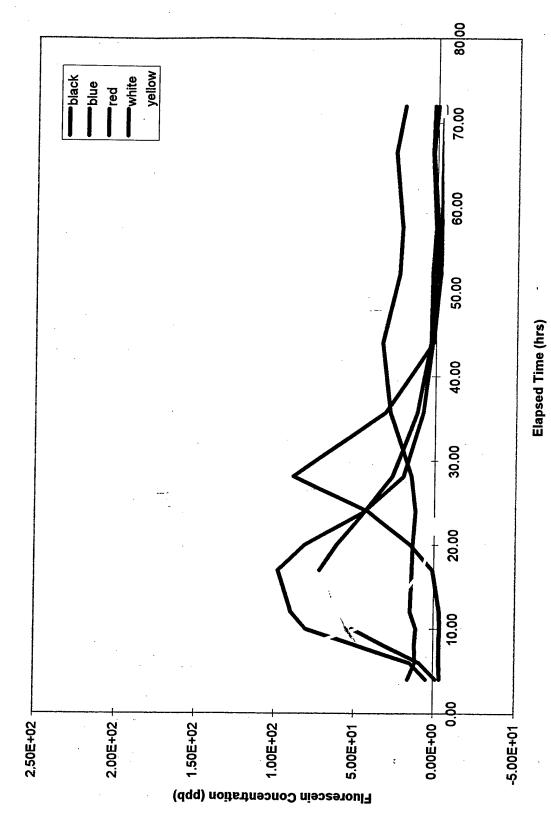


Figure B-9.

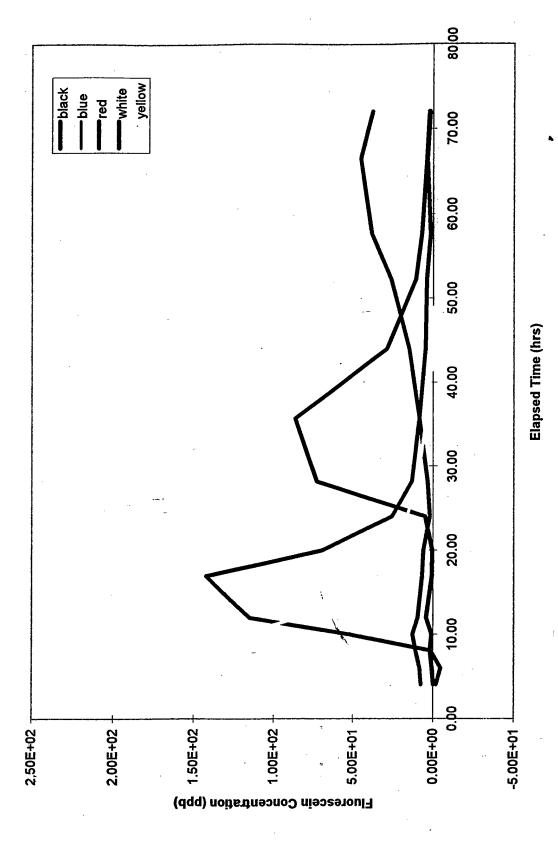


Figure B-10.

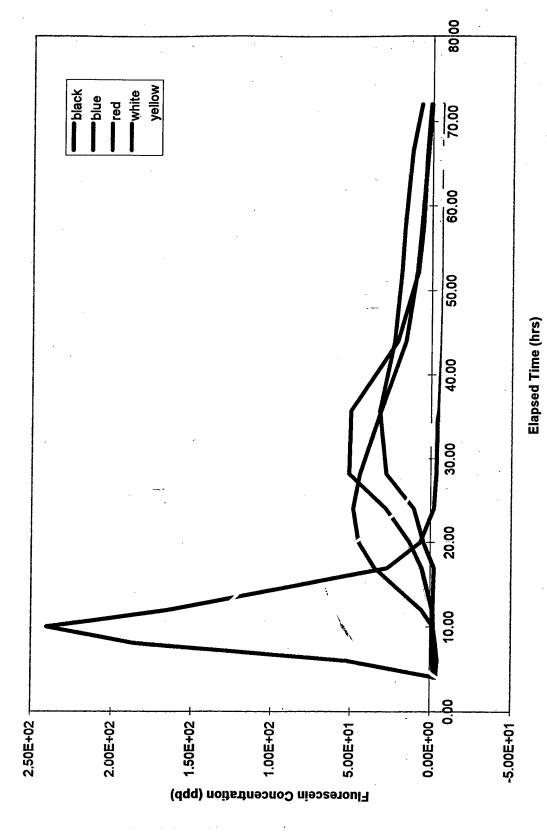


Figure B-11.

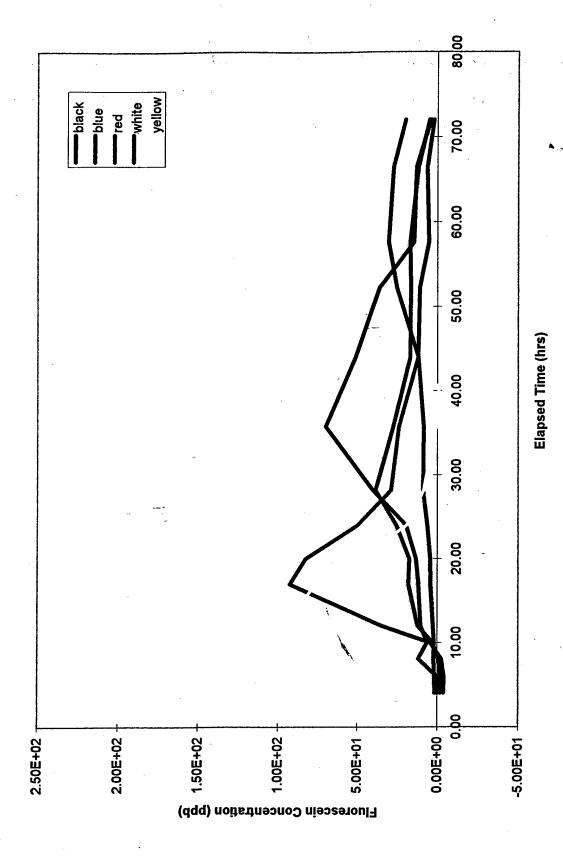


Figure B-12.

### APPENDIX C

### TRACER SOP

STANDARD OPERATING PROCEDURES
FOR
NON-HALOGENATED VOLATILE ORGANICS
(TRACER ALCOHOLS)
BY GAS CHROMATOGRAPHY
MODIFIED SW846 METHOD 8015

# STANDARD OPERATING PROCEDURES FOR

# NON-HALOGENATED VOLATILE ORGANICS (TRACER ALCOHOLS) BY GAS CHROMATOGRAPHY MODIFIED SW846 METHOD 8015

Draft October 6, 1995 Revision 1- October 12, 1995

# 1.0 Scope and Application

1.1 This method is used to determine the concentration of nonhalogenated volatile organics (tracer alcohols) in water samples. The analytes to be analyzed by this method are methanol, n-pentanol, n-hexanol, and 2,2-dimethyl-3-pentanol. The purpose of this SOP is to ensure reliable and reproducible analytical results of the tracer alcohols in water samples for on-site or laboratory-based Gas Chromatograph (GC) analyses.

# 2.0 Summary of Method

- 2.1 This method describes the analytical procedures and the general gas chromatographic conditions necessary for the detection of tracer alcohols used as partitioning tracers. Samples are analyzed by direct injection. Detection is achieved using a flame ionization detector (FID) and the gas chromatograph is temperature programmed to separate the organic compounds of interest.
- 2.2 The gas chromatograph is calibrated using a five point calibration curve for the tracer alcohols of interest. Verification of the instrument stability is checked every ten samples with a mid-point calibration standard. The method has been found to provide reliable and reproducible quantitation of alcohol tracers for concentrations >1 g/ml. This value will be verified in a method detection limit (MDL) study according to Fed Reg. 40 CFR, Part 136 every six months. The method detection limit study encompasses analyzing seven standard replicates (concentration at three times the expected MDL), calculate the standard deviation and the MDL is three times the standard deviation.

# 3.0 Sample Containers, Collection, Transportation and Storage

3.1 Sample Containers. Water samples are contained in 40 ml (Certified clean) glass amber vials with Teflon-faced septa caps.

- 3.2 Sample Collection: each sample vial is completely filled with aqueous sample, such that no headspace of air exists, and capped. The vials are not opened until the time of analysis. All vials must be labelled with location, date and time of collection, and technicians initials. In addition proper chain-of-custody documentation must accompany all vials.
- 3.3 Transportation and Storage: for field studies the samples are stored in coolers containing blue ice, and later stored in refrigerators in a trailer located on the site. Samples may be subjected to on-site GC analysis, and/or shipped to another analytical laboratory. Those samples shipped to a fixed laboratory are packed in coolers and shipped via overnight air express (e.g., FED Ex). The samples are stored in the cold storage room or refrigerator at 4 C until they are ready for GC analysis. After analysis, the samples are returned to cold storage. For laboratory studies, the samples are stored in a refrigerator if the period prior to analysis is expected to exceed eight hours. Samples must be analyzed within 7 days from collection unless chemically preserved with H<sub>2</sub>SO<sub>4</sub> to a pH <2 which allows for analysis within 6-weeks of collection. Holding blanks will be maintained during the holding period and analyzed to determine the extent, if any, of biological degradation.

# 4.0 Apparatus and Materials

4.1 Gas Chromatograph System: Hewlett Packard HP5890 Gas Chromatograph, this GC system is capable of temperature programming and has a flow controller that maintains constant column flow rate. The system must be suitable for on-column injections, and all required accessories including an FID detector, a packed or split/splitless injection port, and an autosampler. A data system for measuring peak area and/or peak height for data acquisition and processing is essential.

# 4.2 Gas Capillary Columns:

Capillary column: Supelco SPB  $\stackrel{*}{>}$  column 30m  $\times$  0.32mm i.d, 0.25 m (phase film)

( = 320) (separates mostly by bp) or equivalent. (J&W, DB-5; Restek, RTx-5)

or

Capillary column: Supelco Supelcowax 10 column  $30m \times .25mm$  i.d, 0.25 m (phase film) ( =250) (separates mostly by polarity) or equivalent

(J&W DB-WAX; Restek, Stabilwax)

The capillary column chosen must meet the needs of the analysis and does not introduce contaminants which interfere with the identification and quantitation of the compounds of interest. Caution should be taken in choosing a column as introduction of water onto a column may damage it. Manufacture's recommendations will be taken into account for column selection.

- 4.3 Gases: Zero-grade air and ultra-high purity hydrogen are used for the FID, and ultra-high purity helium is used as the carrier gas.
- 4.4 Glassware: Syringes (sizes to be determined), Class A volumetric pipettes (1 or 2 ml) required for sample dilutions, Auto sampler vials with Teflonfaced caps, Volumetric Class A pipettes (0.5, 1, 2, 5, 10 ml) are required for the preparations of the calibration standards.

## 5.0 Reagents and Standards

5.1 Reagent water: Reagent water is defined as a water in which an interferant is not observed at the method detection limit (MDL) of the analytes of interest.

#### 5.2 Standards

- 5.2.1 Analytical standard solutions are prepared from pure standard materials or purchased as certified solutions. (??Source??). Stock standard solutions, at 1000 g/ml of each analyte, are prepared in reagent free water and kept in glass vials with Teflon-lined caps; minimal headspace ensures no volatile losses. The stock solutions are stored a -10 C to -20 C and should be protected from light. The solutions must be labelled accordingly: date prepared, preparer, concentration, batch#, and expiration date. Standards must be prepared every month or sooner if comparison with the Quality Control Standard or check standard indicates a problem.
- 5.2.2 Working Stock Solution: prepare a working stock solution by a one fifth dilution of the stock standard (1000 g/ml) to a concentration of 200 g/ml.
- 5.2.2 Working Calibration Standards are prepared by diluting the stock standard solutions in reagent water. Prepare five calibration levels as follows: the low standard is prepared at the reporting limit of the method, four standards are prepared by dilution of the working stock solution (e.g. 200 g/ml), while the fifth is an undiluted solution. This gives five calibration levels (g/ml) as follows

5	Level 1	Level 2	Level 3	Level 4	Level
Analyte Concentration	0.99	9.52	18.18	100.0	200.0

#### 5.2.3 Initial Calibration Analytical Sequence

VIBLK
Alcohol Level 1 Standard
Alcohol Level 2 Standard
Alcohol Level 3 Standard
Alcohol Level 4 Standard
Alcohol Level 5 Standard
VIBLK

VIBLK (Volatile instrument blanks) are analyzed to demonstrate that the system is free of contaminants which may interfere with the analysis. A blank is a injection of reagent free water. The VIBLK must not contain target analytes at or above the reporting limit.

- 5.2.4 Linearity Requirements: The calibration curve must have a correlation coefficient (r) 0.995 using linear regression for quantitation to be performed.
- 5.2.3 Continuing Calibration Check Standards (CC): A calibration check standard is prepared at the mid-point concentration (Level 3). In order to ensure instrument stability the CC is analyzed at a minimum of once every 10 sample injections (not including calibration standards or instrument blanks) and at the close of an analytical run. In order to continue analysis the CC standard must meet the following quality control criteria:

The percent difference (%D) between the calculated concentration and the nominal concentration must be 15.0%.

%D =  $\underline{\text{Conc. (nominal)} - \text{Conc. (calc.)}}$  x 100 Conc. (nominal)

The retention times of the tracer alcohols must fall within the established retention time windows (See Section 6.3).

# Continuing Calibration Analytical Sequence

inj.	<u>Lab ID</u>
1-7	Initial Calibration
8	Quality Control Sample
9-19	Samples
20	VIBLK
21	CC alcohol standard Level 3
22-32	Samples
Closing	VIBLK
	CC alcohol standard Level 3

If the above QC criteria are not met, standards and VIBLK's may be injected a second time. If they fail to pass the criteria again, the analysis must end. The GC system must be inspected for problems to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with the sample analysis. All samples that were injected after the sample exceeding the criteria must be reinjected.

5.2.4 Quality Control Standard: A quality control sample (QCS) prepared from an independent source other than that of the calibration standards must be analyzed after the initial calibration, to ensure proper instrument calibration and quantitation. The recovery limits for the QCS should be 70-130%. If the QC recovery limits are not met, the GC system and the standard preparation documentation must be inspected for problems to determine the cause and perform whatever maintenance is necessary before recalibrating and proceeding with the sample analysis. All samples injected after a QCS that fails must be reinjected.

#### 6.0 Instrumental Procedures

- 6.1 Gas Chromatographic Configuration
  - 6.1.1 A capillary column chosen to meet the needs of the analysis is installed into FID of the HP 5890 GC system.

# 6.2 Recommended operating conditions are as follows:

Injection port temperature 230 C
FID detector temperature260 C
Initial column temperature 40 C
Initial hold time 1 min.
Ramp rate 25 C/min.
Final column temperature 200 C
Final hold time 2 min.
Carrier: helium, 20-30cm/sec (set at 40-60 C)

# 6.3 Retention time windows (Reference SW846 Method 8000, Section 7.5)

Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of a mid-level standard containing all compounds of interest throughout a 72-hour period. Note: serial injections over less than a 72-hour period result in retention time windows that are too tight. Calculate the standard deviation of the three absolute retention times for each analyte. Plus or minus three times the standard deviation of the absolute retention times of each standard will be used to define the retention time window; however the experience of the analyst would weigh heavily in the interpretation of chromatograms. The laboratory must calculate new retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

# 6.4 Gas Chromatographic analysis:

- 6.4.1 Sample preparation: Water samples are received in 40 ml vials. An aliquot of the sample is transferred from the sample vials after they reach ambient temperature to the GC autosampler vials (size, use manufacturer suggestion) using an analyte free sub-sampling device (i.e. syringe). The vial is capped and properly labelled for placement in the autosampler tray.
- 6.4.1 Direct Injection: 1.0ul of the sample is injected into the GC using an automatic sampling device. Sample injections are properly documented in the analytical runlog.
- 6.4.2 Sample dilutions: If the responses for any analyte exceed the linear range of the system, dilute the sample and reanalyze. Use the results from the original analysis to determine the appropriate dilution required to get the largest analyte peak within the upper half of the calibrated range. Quantities used must be documented.

- 6.4.3 Contamination by carryover can occur whenever high level and low level samples are sequentially analyzed. To reduce carryover, the injector syringe will be rinsed with reagent water three times between analyses. A sample analyzed after a sample with a response outside the calibrated range will be inspected for carryover and reanalyzed if carryover is suspected. If a sample is anticipated to be of a level close to exceeding the calibration range, an VIBLK should be methodically placed in the autosampler tray.
- 6.5 Analyte Identification: Analyte identification is based on absolute retention time as established in Section 5.3. Establish daily retention time windows for each analyte using the absolute retention time for each analyte in the initial mid-level CC standard for that day. The daily retention time window equals the retention time ± three times the standard deviation determined in Section 5.3.
- 6.6 Analyte Concentration: When an analyte has been identified, the concentration will be based on peak area, which is converted to concentration using the linear standard calibration curve (External standard calibration).

Concentration (ug/ml) = Calculated conc. (ug/ml) from calibration curve \* D.F.

# D.F. = dilution factor = <u>Final diluted volume</u> Volume of sample added

# 7.0 Quality Control

7.1 GC injector septa must be changed every 60-80 injections or sooner if any related problems occur (i.e. spray back after sample injection, retention time shift, calibration standard response low).

Called a still at the

- 7.2 Injector liner must be cleaned or changed every 60-80 injections or sooner if any related problems occur (i.e. increase base line, noise, integration or signal deviations).
- 7.3 A VIBLK must be analyzed every ten samples and before the closing CC standard. This blank is prepared in the same manner as the standards. In order for the VIBLK analysis to be acceptable the blank must not have any target analytes detected at or above the reporting limit. If a VIBLK fails to meet this criteria, all samples associated with this blank must be re-analyzed or analysis should not continue until an acceptable blank is analyzed.
- 7.4 A method blank (VBLK) must be prepared with each analytical batch. The VIBLK is distinctly different from the method blank in that it is prepared at the same time as the calibration standards whereas the method blank is prepared

with the samples, i.e, vial is filled for method blank same time and place as samples. In order for a VBLK to be acceptable the blank must not have any target analytes detected at or above the reporting limit. If a VBLK fails to meet this criteria, all samples associated with this blank must be re-analyzed or analysis should not continue until an acceptable blank is analyzed.

- 7.5 Initial standard calibration must be conducted every time the flame is started, daily, or for each analytical batch, whichever is more frequent.
- 7.6 Each analytical sequence, the continuing calibration check standard should be evaluated (Refer to Section 4.0) to determine if the chromatographic system is operating properly. Careful examination of the standard chromatogram can indicate whether the column is still good, the injector is leaking, or the injector septum needs replacing. If any changes are made to the system (e.g. column changed), and the CC standard does not met the continuing calibration criteria as outlined in Section 4.0, recalibration of the system must take place.

## 7.7 Matrix Spike/Matrix Spike Duplicate Analysis

7.7.1 An MS/MSD is required for every 20 samples or portion thereof. The MS/MSD analysis consists of spiking two aliquots of the same sample prior to analysis.

Matrix Spike Compounds	s Amount Spiked	Recovery	Limits RPD
methanol	18.8 ppm (TBD)	TBD	- 198 <b>0-350</b> Hass
n-pentanol		Ħ	
n-hexanol	•		
2,2-dimethyl-3-pentanol		H .	

TBD = To be determined

Calculate each percent recovery (%R) as follows:

%R = (Spiked Sample conc. - Unspiked Sample conc.)/Conc. of the spike \* 100

Calculate Relative Percent Difference as follows: RPD = |MS %R - MSD %R| \* 100 ½ (MS%R + MSD %R)

Recovery limits (established control limits) should be established when sufficient data points (20 sample analyses) have been collected. Once a minimum of 20 samples of the same matrix have been analyzed, calculate the average percent

recovery (p) and standard deviation of the percent recovery (s) for each of the analytes. Calculate the Upper and lower recovery limit for each compound as follows: Upper Control limit (UCL) = p + 3s, Lower Control Limit (LCL) = p - 3s. Refer to SW846 Method 8000A, Section 8. If the recovery of any matrix compounds is outside the established limits the deviation will be investigated and the sample analysis thoroughly reviewed to determine if corrective actions should be taken.

# 8.0 Compound List and Reporting Limits

- 8.1 Compound List given in Section 1.1
- 8.2 Reporting Limits

Compound reporting limits as established from the Method Detection Limit Study are (3 x MDL). Reporting limits are also evaluated based on the chromatography of the compound and the sensitivity of the compound. The reporting limit as determined from previous analysis of this method has been 1ppm (ug/ml).

#### APPENDIX D

#### SOIL CONCENTRATION DATA

Final Pre-Treatment Soil Chemical Analytical Data

Michigan	Technological	1 lakombi.	1								i			
Report Da	Report Date 1/9/96, Sample analysis: 11/21/95 - 12/05/95	mple analys	sts: 11/21/85	12/05/95	1	-	-					:	;	
Analyte C	Analyte Concentrations = mg/k	ns = mg/kg	dry soil	-									+	
			Concentration	Concentration in Coll (marker)	10/4									
Sample ID Sample ID	Boring ID	decthilleen	To and	The Control		-	,							
-		12-13	0.0000	00000	0000	00000	8	o-xylene	m-xylene	decane	de la	undecene	8	ğ
22	U1 2722	13-14	0.0000	0.0000	0.0000	0.0000	00000	0000	1188	0.9002	0.0321	2.8058	0.3448	0000
	U1 2722	14-15	0.0000	0.0000	0.0000	0.2007	0.0267	0.0281	0.1640	2.3987	1 0000	7 80347	4 7932	0000
	U1 2722	15 82	0.0000	0.0000	0.0000	0.3491	0.0877	0.2977	0.2401	4.6148	0.4801	12 2213	1.7510	0.0192
	012722	18-17	0.1270	0.0211	0.0293	1.5092	0.5509	2.6767	1.4283	37.2139	3.0391	94 8138	0.4801	2000
100	22.25	17-18	0.9656	0.000	0.1264	6.5668	2.8162	14.1948	5.2631	109.6890	8.2032	238.9844	26.9430	61158
0	27.22	18-19	4.5363	0.0435	0.0182	13.9535	4.5271	22.5291	7.6181	67.0914	9.2607	132.3361	42.7530	6.7640
66	111970	30.00	0,000	0.000	0.0000	9.0584	2.9956	15.3212	5.0908	50.9699	6.2065	115.1418	39.5880	0.0393
1010	U12722	22.23	0.2877	28100	8000	5.3232	1.8025	9.1412	3.0735	35.0546	4.1332	93.9932	28.0078	4.1823
1111	U12722	2.5	3.3548	0.0372	2000	0.5170	0.10/2	4884	0.1774	4.4325	0.2484	15.7450	2.8381	0.4470
12 12	U1 2722	23-24	0.000	0000	0000	0000	7777	0000	1.8287	35.6512	22701	16.4293	15.7626	2,4066
13 13	U1 2722	24-25	0.0449	0.0000	0.0000	0.0587	0.0568	0.0604	00000	00/70	4000	8102.1	0.3916	0.1228
14 14	U1 2722	25-26	0.0564	0.0000	0.0000	0.0000	0.0511	0.0552	00000	0.3520	0.0343	1.6340	0.5740	0.2275
15 15	U1 27/22	27-28	0.0000	0.000	0.0000	0.2221	0.0211	0.0818	0.0324	0.2414	0.0306	1 0886	0.500	0.190
16 16	012722	82	0.000	0.0000	0.0000	0.4438	0.0485	0.1568	0.0599	0.0000	0.0812	0000	2000	2000
17 (MW)	012722	2	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	00000	00000	0000	000
222	10.2724	12-13	0.0000	0.0130	0.0000	0.0161	0.0059	0.0075	0.0217	1.6748	0.0215	3.3021	4.1241	0003
222	11 2724	2 2	0000	0.0116	000	0.0221	0.0065	0.0072	0.0330	1.0089	0.0157	4.4566	0.9985	0.0191
24.24	111 2724	15.48	0.000	900	888	0.1096	0.0730	0.3064	0.2022	3.6216	0.2125	9.5318	1.0504	0.0618
25.25	U1 2724	15.5	2000	3000	0.000	0.479	0.4746	2.4960	1.3978	30.2871	2.9505	68.3201	8.7579	1.5885
28.28	U1 2724	17-18	00171	0000	92.00	0.1001	3.3032	16.5811	6.1620	128.5176	9.1670	215.3111	28.9903	6.8127
	U1 2724	18-19	1.8384	0.010	0.00	0.3032	0.1945	0.8902	0.7983	22,3906	1.3874	63.2975	6.8364	0.0133
	U1 2724	19-20	3.5701	0.0270	0.0143	6 6818	23820	10 4798	8000.7	23/36	9.6185	110.3897	28.3007	5.8118
	U1 2724	20-21	1.5840	0.0262	0.0417	4.0826	1 9893	9 8228	3 5590	89 9977	5.4600	1082335	27.2314	4.5588
	U1 2724	21-22	6.1022	0.0263	0.1531	5.0562	1.9552	9.1540	3.0598	67.2393	3 0000	1/3/05/07	20000	6.1402
	U1 2724	22-23	1.6729	0.0262	0.0309	1.4869	0.6303	2.8902	0.9724	312321	1.2968	53 3283	11 1000	4024
1	U1 2724	22	0.2043	0.0214	0.0000	0.2953	0.1676	0.7913	0.2867	19.5421	0.4382	10.3214	5 9718	0000
l	1 2741	12-13	0000	0.0211	0.0296	0.0161	0.0046	0.000	0.0693	2.7269	0.0098	13.1962	0.8788	0.0265
	11 2741	14.15	3000	0000	0000	0.0117	0.0069	0.0084	0.0710	22476	0.3037	10.2154	1.8852	0.0253
	U1 2741	15-16	0.000	00000	3000	0.0104	0.0000	0.0000	02224	2.7256	1.8746	14.4138	3.9055	0.2490
37.37	U1 2741	16-17	0.2231	0.0118	0.0082	3.0562	1.0749	6,700	9 5528	12.2192	8.0909	57.9591	7.2535	0.2197
	U1 2741	17-18	0.5359	0.000	0.0000	3.9806	1.6912	8.4250	32082	66.0839	5.4930	82 601B	17.001	2.1529
	U1 2741	9-19	0.7714	0.0168	0.000	6.5630	2.6514	12.7389	4.7577	12,7389	4.7577	94 9622	18 5672	3 5060
	14 2741	200	0.0000	0000	000	6.4751	1.9042	9.6941	3.2858	38.2468	4.5768	89.7012	29.9508	4.3535
	111 2741	3 20	1.7/40	0.0138	0.000	3.3839	1.1700	5.5386	1.8641	40.5178	2.6536	21.3657	19.8958	2.9170
Γ	U1 2741	22.22	1.50	386	0000	2.7702	1.0207	4.8357	1.6427	38.0548	2.3780	114.4737	22.6206	3.0565
	U1 2742	12-13	0.000	0000	0000	0000	0.0323	0.0343	0.000	0.3666	0.0252	0.6365	0.2815	0.0732
	U1 2742	13-14	0.0000	0.0000	00000	0000	00000	0.0272	0.000	0.7475	0.0419	3.0622	0.2745	0.2086
	U1 2742	14-15	0.0000	0.0000	0.0000	0.0652	0.0117	0.0168	0.0675	0.704.0	0.07	0.0982	0.0954	0.0091
8	U1 2742	16-17	0.1323	0.0196	0.0306	2.0578	0.8931	4.7757	2.2457	35249	3 0450	3.0234	0.1822	0.0102
88	U1 2742	15-16	0.0156	0.000	0.0000	0.7634	0.2055	1.0441	0.5358	9.0038	0.6959	22.8802	2 1385	0.2456
21 8	2/42	18-19	1.7659	0.0225	0.00	8.9742	3.3037	15.3018	5.6687	103.6526	8.3982	29.9938	23.1104	4.3537
280	U1 2742	20.2	4 1880	0.0638	0.0285	19.7116	5.8755	28.9245	9.9573	82.3801	12.6405	154.4826	58.3095	8.1941
150	U1 2742	23-24	0.1343	0000	2000	3.3007	2000	4.1639	4.8753	89.8363	10.3371	179,3142	38.8379	7.2411
					173333	V. 10601	0.0244	0.1035	0.0410	1.1265	0.0507	3.0251	0.7337	0.1459

Final Pre-Treatment Soil Chemical Analytical Data (Continued)

	. !			٤	0.0124	0.0591	0.0467	0.0414	0.7306	3.7555	8.1612	8.4089	22566	0.0000	0.000	0.0438	0.3526	3.0618	4.6004	3.4615	4.2059	0.3514	0.0196	0.0140	0.0032	0.0072	0.0476	7.1007	6.9819	3.1060	3.1152	8.8639	0.5178	0.1012	0.0218	0.0587	0.0361	0.0381	4.5120	4.5591	8.7538	6.6802	5.4443	2,9116	7084
				g	0.0229	0.1492	0.0707	0.0892	3.4700	14.4836	41.1891	77.4084	12,2908	0.0689	0.1673	0.1975	1.5443	13.6452	24.2783	20.7074	30,2325	2.0936	81.1551	15.5915	5.0753	0.5849	6.4089	63.1726	41.4458	16.3246	18.4197	48.5173	2.9818	0.7037	200	222948	8012	4100.00	25.8485	18.9488	38.6381	32.0727	28.4670	17.6775	11.3977
				Undecene	0.0466	8.3577	0.1312	2.3930	31.8249	150.7700	145.9509	268.3261	80.7037	0.6784	1.1893	0.2419	22.2392	225.9438	112.1010	88.3518	138.9689	11.117	2.2645	0.5124	0.9347	1.4633	5.5952	289.1504	214.2280	232.5786	95.1490	252.9410	2027	00,00	0 5570	0.0073	4.3/00	246	313,2309	8/01/02	313.6274	159.7864	164.1147	149.2668	8188.17
				OE C	0000	0.0716	0.0456	0,000	1.0226	6.0528	12.3177	9.6088	1.3704	0.000	0.0000	0.0000	0.4635	5.3833	2.8097	2.6827	3.5163	0.1787	0.0430	0.0000	0.0000	0.000	0.1069	16.4959	6.6723	5.7376	2,4257	14,4920	1 1000		0000	2000	00000	7.8474	7050	16 1050	10.1030	4.0580	5.6987	2.4803	2362
				0 4440	0.143	220/0	0.4134	0.4812	10.8367	74.2899	80.3813	82.0064	21.7340	0.1255	0.4657	0.9523	9.0538	73.8627	41.6205	21.6908	54.3381	2.9342	1.1675	0.9426	0.3247	0.3715	32082	105.3288	61.5778	13.4901	81.35.14 444 6004	11:000	2.304R	0.4179	1 0307	1.1585	32251	118 0713	110 027R	110 5526	44 6054	41.5257	45.2515	42.0051	24.0450
			1	0000	385	0.0250	0.0188	0.000	0.6671	3.0459	8.9709	7.4078	1.0293	0.0000	0.0000	0.000	0.3413	3.4755	2.1334	1.9502	2.7916	0.1182	0.0384	0.0091	0.0132	0.0145	0.1449	9.9748	4.8309	0.0030	1.3030	0.2060	0.023	0.0000	0.0000	00000	0.0917	3.4703	5.8005	11.3702	2000	2 0404	1 0074	00000	0.000
			Cardena	0.000	0.0186	2000	2000	0000	27.70	201.00	25.4748	22.1233	3.0483	0.000	0.0000	0.000	1,567	0.018/	6.2808	2.9630	8.4163	2000	0.0067	0.0063	0.0082	0.000	0.0348	0770	12.3147	2,6340	19.6577	0.5729	0.0521	0.0000	0.0000	0.0000	0.1106	4.1938	12.0886	31.3799	7 1910	11 0801	5.8110	2.5899	00090
		•	8	0.0000	0.0183			2000	4 4697	1305	4.9094	4.3303	0.0000	3000	0000	0,000	0.1235	/out.	1808.	7070	1810.1	0.0720	4/000	0,000	0.000	0.00483	0.0132	0.000	1 0679	8028	4.1327	0.1219	0.0115	0.0000	0.0000	0.000	0.0341	0.8443	2.7153	5.9337	15081	2 1024	1.2713	0.5392	0.1421
			tolvene	0.0049	0.0118	00184	00423	0 74 80	3 6020	44 5000	12 8246	0 4007		388	3000	0.000	2 8458	00000	00000	5.50023	20,000	00467	0.0167	350	2000	97200	4 4450	7 2201	2.1484	1.7871	10.1177	0.3398	0.0269	0.0000	0.000	0.0000	0.0936	0.5771	3,4108	12.9009	3.8575	5.0805	2.8977	0.0000	0.1779
		g/Kg)	\$	0.0000	0.0000			l	Ĺ	l			1		l	-				l						OESO O		0000	00000	0,000	0.0622	0.000	0.0000	0.0000	0.0000	0.0000	0.0859	0.0000	0.1147	0.0132	0.000	0.0000	00000	0.0000	0.0000
MI 12/05/05		Concentration in Soil (mg/Kg)	berzene		0.000						0000	0.0091	00000	00000	00000	0000	00000	0.0055	0.0118	0.0317	0.000	0.0000	0.000	0.0074	0.0117	0.0230	0.000	0.000	0.0000	0.0000	0.0508	0.0000	0.0000	0.0000	0.0000	0.000	0.0000	0.0000	0.0080	0.0180	0.0000	0.0159	0.0120	0.0000	0.0000
Houghton, MI sir: 11/21/95	dry soil	Concentrati	Z	0.0000	0.0000	0.0000	0.0510	00000	0.6896	0.0000	0.0000	1.3323	0.0000	00000	00000	0.0443	0.9020	0.3986	1.8217	3.7361	0.1266	0.0000	0000	00000	0000	0.0000	00000	1.6352	0.3981	00000	5.6084	0.2169	000	000	0.0000	0000	0.0054	0.1165	0.6562	22485	0.0000	1.8489	2.6748	0.000	0.1232
d University,	ns = mg/kg		depth(feet)	12-13	13-14	14-15	15-16	16-17	17-18	18-19	19-20	21-22	12-13	13-14	14-15	15-16	16-17	20-21	21-22	22-23	23-24	12-13	13-14	14-15	15-16	16-17	17-18	18-19	19-20	20-21	21-22	22-23	723-24	12-13	13-14	12	2	16-17	17-18	<del>8</del>	19-20	20-21	21-22	22-23	23-24
Michigan Technological University, Houghton, MI Report Date 1/9/96, Sample analysis: 11/21/85 - 12/05/95	Analyte Concentrations = mg/kg dry soll			U1 2743	U1 2743	U1 2743	U1 2743	U1 2743	U1 2743	U1 2743	U1 2743	U1 2743	U1 2744	U1 2744	U1 2744	U1 2744	U1 2744	U1 2744	U1 2744	U1-2744	U1 2744	U1 2751	U1 2751	U1 2751	U1 2751	U 2751	01 2751	20/25	8/25	2753	012753	26/25	012753	01 2753	U1 2753	U1 2753	U1 2753	U1 2753	U1 2753						
Michigan Report Dg	Analyte C		Sample ID Sample ID	\$ 5	8	99 99	29 29	88 88	69 69	20 20	71 71	74 74	78 78	79 79	90 80	31 81	82 82	33 83	94 84	98 96	37 87	39 89	90 90	11 91	92 92	ଓଷ	184	95 95	96 96	97 97	88 88	88	30,00		30,00	50.00	20100								113 113
	ļ		Sample										•	•	3	<b>.</b>	3	3	3	3	3	8	8	8	6	6	8	6	°	<u></u>	6	5	2 \$				2   3			<b>≦</b>  \$	ğ	٤	٤		ِ ا

Final Post-Treatment Soil Chemical Analytical Results

HillAFB post-treatment analysis   HillAFB post-treatment analysis   Heport date: 12/13/96, Sample analysis: 11/23/96 - 11/26/96   Analyte Concentrations = mg/kg Soil	sanalysis   Sample analysis	11/23   11/23	23/96 - 11/26, 23/96 - 11/26, 23/96 - 11/26, 23/96   11/26, 23/96	0.004	toluene								
1300 Sample and 1300 Sample an	Olean Parabysis Digital Paraby	1123 1000 1000 1000 1000 1000 1000 1000	896 - 11/26 enzene (c. 0.054 0.005 0.007 0.008 0.006 0.006 0.006 0.006 0.006 0.006	0.004									
19 10 depth 1-15 17-18 18-19 1	(feet) tca	0.000 bdd 0.000	0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005 0.005	0.004									
20 C C C C C C C C C C C C C C C C C C C	((eet)) (ca	0.00 bd	0.005 0.005 0.005 0.006 0.006 0.006 0.006 0.006 0.006 0.006	0.004									
9 1D depth 1-12 12-13 12-13 12-13 13-13 12-13 13-13 12-13 13-13 12-13 13-13 12-13 13-13 13	Beet) (ca		4 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	0.004									
9 ID depth 1-12 1-1-13 1-1-17 17-18 1-18 18-19 2-22 22-23 2-23 22-23 2-24 12-25 2-25 22-23 2-25 22-	(1) (1) (1) (1) (1) (1) (1) (1) (1) (1)		4 5 9 5 5 5 5 5 5 5 5	0.0047	П								
99 U Gepting 10 Control of Contro	9e()(ta		4 2 8 2 8 2 8 2 8 2 8 2 8 2 8 2 8 2 8 2	0.004					_				
2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	<del></del>	0.005 0.005	0.054 0.006 0.006 0.007 0.006 0.006 0.006 0.006 0.006	0.004		8	o-xv-l	Ž.	decane	4 5		T	
20 20 20 20 20 20 20 20 20 20 20 20 20 2		0.002 0.0152 0.0152 0.0152 0.005 0.005 0.006 0.006	0.000 0.000	0.002	0.021	900.0		0.005	0.082	2	or degrie		nap
24 24-25 22-23 22-		0.00 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000 0.000	0.005	0.008	0.001				3 6	0.030	0.00	000
20 20 20 20 20 20 20 20 20 20 20 20 20 2	<del>                                     </del>	0.000 0.000	0.000 0.000 0.000 0.000 0.000 0.000 0.000	0000	0.020	0.073	l			305	200.0	0.020	0.031
202 22 22 22 22 22 22 22 22 22 22 22 22		0.005 0.005 0.005 0.005 0.005 0.005	0.006 0.008 0.008 0.006 0.006	0.030	0.056	0.00	l			000	76.03	3.839	1.843
22 22 23 23 24 25 25 25 25 25 25 25 25 25 25 25 25 25		0.000 bdi	0.00 0.008 0.008 0.008 0.008	0.032	2338	2 705	Γ			0.009	10.269	0.144	0.083
24 24.25 112 12-13 14 14-15 16 16-17 20 20-21 24 24-25		0.000 0.000	0.008 bdid 0.006 0.006	0.028	2 801	2000			1	8.886	323.513	57.702	5.590
12 12-13 14 14-15 20 20-21 24 24-25		0.00 0.00 pdd	0.006 0.006 0.005	010	0.054	24.0	1/5.7	1		3.651	140.743	28.320	4.103
14 14-15 16 16-17 20 20-21 24 24-25		0.09 00 00 00 00 00 00 00 00 00 00 00 00 0	0.006 0.006 0.005	007	2000	2 6	l			0.342	8.503	3.902	0.511
16 16-17 20 20-21 24 24-25		0.040 1000 1000 1000 1000 1000 1000 1000	0.006	500	200	0.002	1			0.001	1.309	0.237	0.129
20 20-21	$\{\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},\{\},$	0000	0.006	7000	0.012	0.002				0.005	0.893	0.042	0.003
24 24-25	111111	0000	0.005	0.024	0.012	0.003		0.00	0.611	0.00	4.322	0.073	0.069
			0.000	0.020	0.609	0.347		0.701	0.480	1.359	0.121	9315	1 630
		$\dagger \dagger \dagger$		2000	0.128	0.080	0.368	0.153		0.332	14.888	2.858	0547
	+ + +	$\parallel$											
	H	$\dagger$		1	1								
	+												
12.13		200	2000										
14 14-15	+	3 3	337	0.0	0.006	0.00		0.002	0.012	0.002	0.379	0.023	0005
16 18-17		3 2	3 3	0.020	2	0.00		0.002	0.023	0.000	0.087	0.00	0.002
17 17-18	-	1000	0.035	200	37	000		0.001	0.015	pđ	0.069	0.004	0.011
18 18-19	ŀ	0.002	3 2	3 2	3 3	500		0.00	0.026	B	0.320	0.004	9000
19 19-20	$\mid$	9000	0.007	3 2	3 2	7000	1	0.003	0.025	0.00	0.216	9000	0.012
20-21	L	0.023	9000	36	0.014	20.00		0.0	0.423	0.00	2.307	0.054	0.00
20/22 20-22	-	Z	0.005	3 3	4 204	0.018		0.878	21.137	1.411	10.631	10.277	1.595
23/2423-24		B	2	900	0.017	2000		1.258	26.645	1.813	17.103	14.286	2.182
23/2423-24	_	큠	Þ	9000	0.015	200		0.023	0.692	0.045	1.738	0.439	0.126
	L	000.0	0000	0000	000	200		0.022	0.688	9	1.740	0.442	0.102
	L	000.0	0000	0000	000	3 8		300	0.000	0.000	0.00	0.000	0.000
22/22 22-23		2002	0.008	0.005	0000	350		0000	0000	0.000	0000	0.000	0.000
24/2824-25		2005	0.008	9000	9000	200	2420	0.020	0.834	0.050	2.262	0.441	0.076
12 12-13	L	.001	0.005	0.007	900	3 6	0.021	0.007	0.115	0.002	0.304	0.137	0.068
14-15		2005	0.006	0.018	2000	300	33.0	0.002	0.026	0.001	0.009	0.003	0.005
16 16-17	-	100	9000	200	330	300	0.011	0.003	0.437	0.00	2.040	0.034	0.007
17 17-18	-	900	9000	010	7	300	0.163	0.002	0.008	0.002	0.058	0.004	0.003
17-18	-	200	0.007	0018	000	3 8	2000	0.002	0.023	0.001	0.200	0.005	0.005
18 18-19	L	B	pq	Ē	0.012	300	277	2000	0.054	0.001	0.184	0.00	0.002
11 21-22	_	025	0.004	0.50	0.050	2000	1000	0.002	120.0	0.00	0.144	0.013	0.00
11 21-22	-	020	9000	0042	000	0.617	50.	0.40	18.728	 86.	8.431	6.667	0.997
2 22-23		8	0.010	000	0.00	0.614	20.0	0.399	19.634	1.249	8.052	6.195	0.883
4 24-25	-	902	B	0.00	0.052	20.00	0.247	0.096	6.121	0.280	11.538	1.817	0.255
4 24-25		83	Ē	0015	000	40.0	0.185	0.027	2.232	0.078	3.694	0.500	0.084
				200	2.0.0	0.0	0.189	0.027	2.259	0.080	3.778	0.507	0.079
	7.783-12 12-13 7.783-16 16-17 7.783-18 18-19 7.783-19 18-20 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-20.22 20-22 7.783-10 16-17 7.784-16 16-17 7.784-17 17-18 7.784-17 17-18 7.784-21 12-22 7.784-21 21-22 7.784-21 21-22 7.784-21 21-22 7.784-24 24-25	12-13 14-15 16-17 17-18 19-20 20-21 20-22 20 20-22 20	12 12-13 0.003 14 14-15 bd 16 16-17 bd 17 17-18 0.001 18 18-19 0.002 19 19-20 0.005 20 20-21 bd 20 20-21 bd 20 20-22 bd 20 20 20-22 bd 20 20 20 20 20 20 20 20 20 20 20 20 20 2		0.003 0.002	0.003 0.007 0.001 0.035 0.002 0.007 0.002 0.006 0.002 0.000 0.000	0.003 0.007 0.014  bdl bdl 0.025 0.002 0.005 0.002 0.005 0.000	0.003 0.007 0.014 0.006 bdl bdl 0.020 bdl 0.020 bdl 0.001 0.001 bdl 0.001 bdl 0.001 bdl 0.001 0.001 bdl 0.002 0.001 0.001 bdl 0.002 0.002 0.002 0.002 0.002 0.002 0.002 0.002 0.000	0.003 0.007 0.014 0.006 0.001	0.003         0.007         0.014         0.006         0.001         0.002         0.002         0.001         0.002         0.002         0.001         0.002         0.002         0.001         0.002         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.002         0.001         0.003         0.001         0.003         0.001         0.003         0.001         0.003 <th< td=""><td>0.003         0.004         0.004         0.004         0.004         0.002         0.012         0.001         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         <th< td=""><td>0,003         0,004         0,004         0,002         0,004         0,002         0,003         0,002         0,003         0,004         0,003         0,004         0,003         0,004         0,003         0,004         0,004         0,004         0,003         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         <th< td=""><td>0.003         0.007         0.014         0.006         0.001         0.002         0.015         0.002         0.037           bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.022         0.023         0.000         0.037           0.001         bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.002         0.003         0.000         0.008         0.001         0.008         0.009         0.008         0.001         0.008         0.001         0.008         0.001         0.008         0.001</td></th<></td></th<></td></th<>	0.003         0.004         0.004         0.004         0.004         0.002         0.012         0.001         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003         0.002         0.003 <th< td=""><td>0,003         0,004         0,004         0,002         0,004         0,002         0,003         0,002         0,003         0,004         0,003         0,004         0,003         0,004         0,003         0,004         0,004         0,004         0,003         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         <th< td=""><td>0.003         0.007         0.014         0.006         0.001         0.002         0.015         0.002         0.037           bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.022         0.023         0.000         0.037           0.001         bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.002         0.003         0.000         0.008         0.001         0.008         0.009         0.008         0.001         0.008         0.001         0.008         0.001         0.008         0.001</td></th<></td></th<>	0,003         0,004         0,004         0,002         0,004         0,002         0,003         0,002         0,003         0,004         0,003         0,004         0,003         0,004         0,003         0,004         0,004         0,004         0,003         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004         0,004 <th< td=""><td>0.003         0.007         0.014         0.006         0.001         0.002         0.015         0.002         0.037           bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.022         0.023         0.000         0.037           0.001         bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.002         0.003         0.000         0.008         0.001         0.008         0.009         0.008         0.001         0.008         0.001         0.008         0.001         0.008         0.001</td></th<>	0.003         0.007         0.014         0.006         0.001         0.002         0.015         0.002         0.037           bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.022         0.023         0.000         0.037           0.001         bdf         bdf         0.015         0.001         0.006         0.001         0.002         0.002         0.003         0.000         0.008         0.001         0.008         0.009         0.008         0.001         0.008         0.001         0.008         0.001         0.008         0.001

Final Post-Treatment Soil Chemical Analytical Results (Continued)

	Γ					Ī		Γ	0.036	0.005	0.002	0.000	1.292	Ī	000	0.007	0.003	0.426	1.027	0.060	0.00	023	0.002	0.002	000	0.151	5
			1	1	 L		L	geu	L			L		*	L	Ŀ		Ŀ	Ŀ								Ĺ
								q	0.852	0.355	0.349	0.00	7.983	#VALUE	0.003	0.005	0.012	1.732	6.091	0.259	0.000	0.095	0.00	0.003	0.003	0.721	6.421
								undecane dcb	0.899	0.035	0.028	0.000	11.367	<b>#VALUE!</b>	0.145	0.056	0.088	9.350	11.370	1.844	0.00	2.082	0.150	0.200	0.144	1.622	72.907
									0.003	0.000	0000	0.00	1.692	#VALUE!	0.002	0.003	0.005	0.270	1.035	0.001	0.00	0.038	0.001	0.004	0.003	0.057	0.825
								decane	0.226	0.049	0.083	000	24.508	#VALUE!	0.004	0.00	0.011	2.311	13.857	0.881	0000	0.728	0.030	0.107	0.023	0.258	1.034
								ı-xx-ii	0.025	0.007	9000	0.000	0.546	*VALUE!	0.003	0.002	0.005	80.0	0.344	0.010	0000	0.00	0.007	0.003	0.003	0.03	0.423
								i/x-o	0.248	0.150	0.147	000	576.	*VALUE!	800	0.183	0.159	0.179	1.162	0.181	0000	0.08	0.011	000	0.114	0.057	1.425
							4	17.0	0.017	0.003	300	300	0.293	#VALUE!	0.002	0.00	0.002	20.03	2 2	200	3 8	0.002	0.002	0.002	0.002	0.014	0.233
							or in	18	0.102	0.00		300	1000	*VALOE!	0.00	000	0.00	0.020	33.0	88	38	3 3	8	0,000	40.0	0.013	3
			98				٤	900	300	200	300				0.022	2000	200	3 3	3 2	800	0.014	200	300	300	6000	3	3
		00/04	11/23/30 - 11/20/36				benzene	10	0000	0000	0000	9000	#VALLE		2000	300	900	3 2	000	000	0.005	2	200	0013	2000	3	3 3
		ahroios 44 K	diyala. 11/2	Soli				0.000	000	1000	000	0.019	#VAI IF	Š	500	000	IIV4	0000	000	0.000	B	Σ	0.00	000	Ξ	E	100
al University	analysis	Sample engine		INS E MG/KG SOII			depth(feet) tca	26-27	27-29	27-29		2		12-13	14-15	17-18	20-21	22-23	24-25		12-13	14-15	16-17	18-19	19-20	22-23	34.95
echnologics	HillAFB post-treatment	Report date: 12/13/96	Anshite Concentration	OTHER MAN			•		2794-28	2794-28		2794-21			2796-14			2796-22			2795-12		1		2795-20		
Michigan T	HIIIAFB por	Report data	Anshab	S DI I			mple ID	38		39 rep			42	43		45						51				55	

#### **APPENDIX E**

# PRE-TREATMENT PITT ANALYSIS PLOTS

From "First Moment Analysis of Hill AFB OU1 Partitioning Interwell Tracer Test Conducted by ARA, April 1996," INTERA, Inc., Austin, TX, and The University of Texas at Austin, Austin, TX, August 30, 1996)

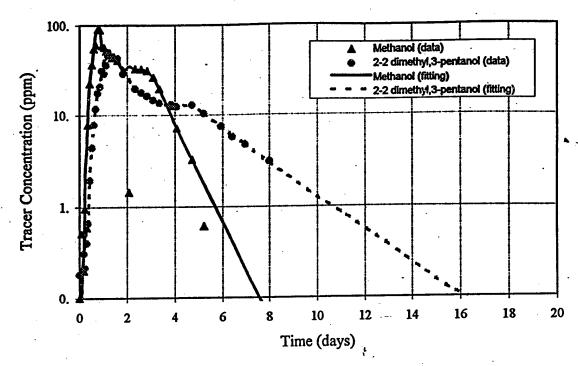


Figure 1a Extraction well 51 tracer response data and corresponding fitting curves

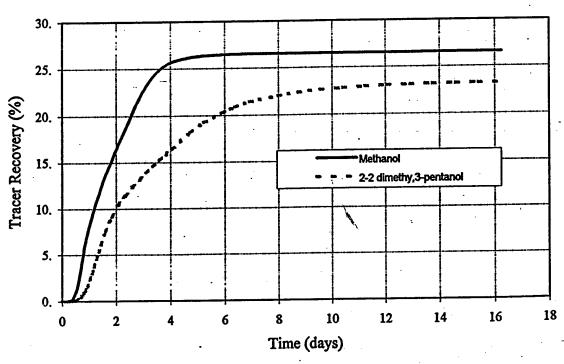


Figure 1b Extraction well 51 tracer recovery

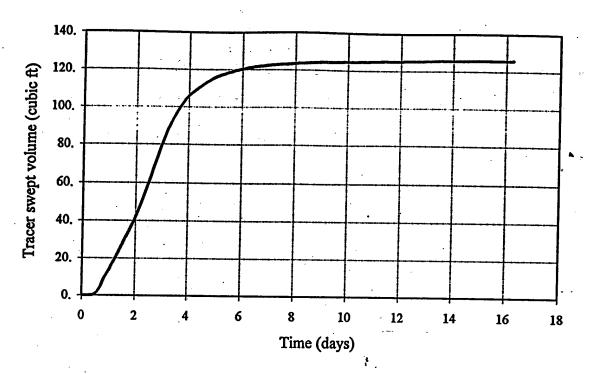


Figure 1c Pore volume swept by tracers capured by extraction well 51

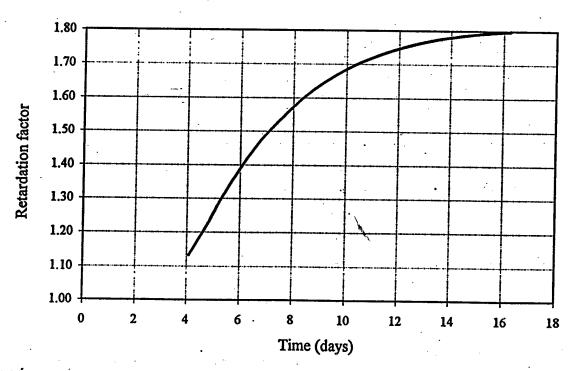


Figure 1d Retardation factor of 22DM3P based on tracer data from extraction well 51

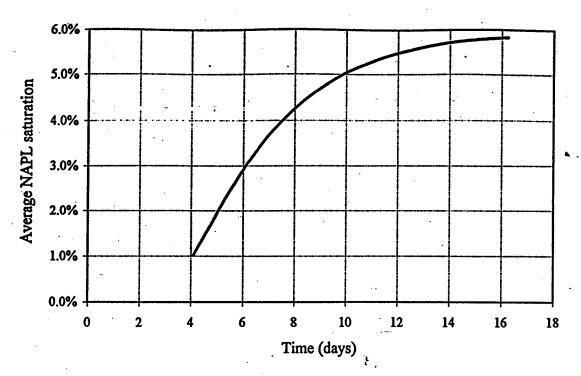


Figure 1e Estimated average NAPL saturation in the swept volume of extraction well 51

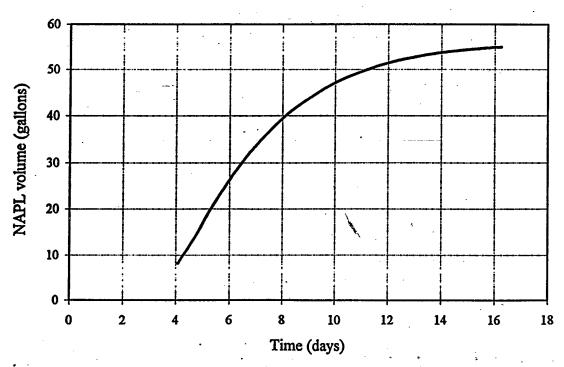


Figure 1f Estimated NAPL volume in the swept volume of extraction well 51

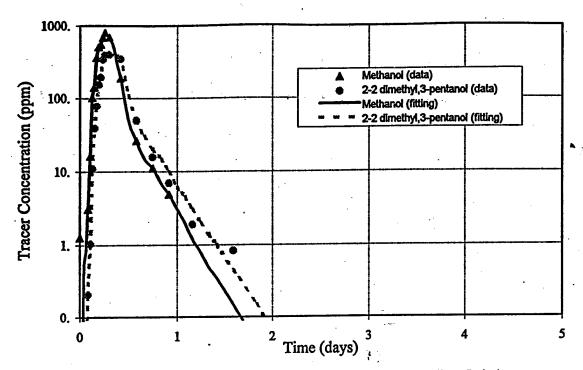


Figure 4a MLS11\_BLACK tracer response data and the corresponding fitting curves

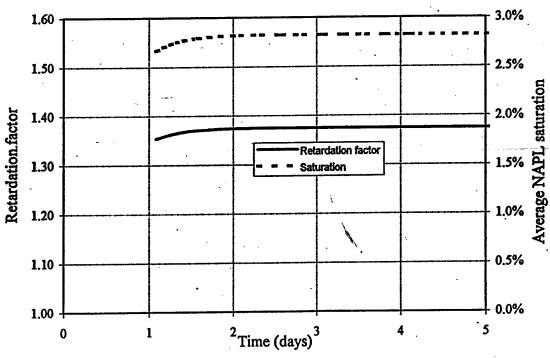


Figure 4b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS11\_BLACK

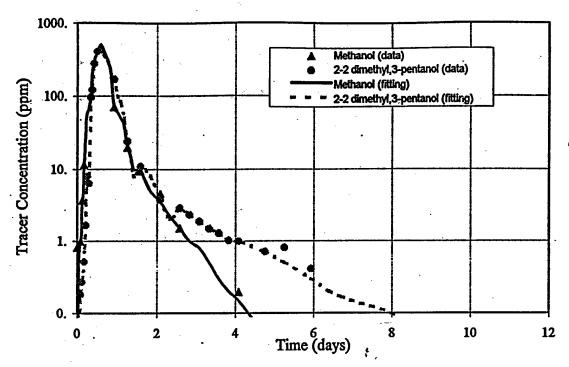


Figure 5a MLS11\_BLUE tracer response data and the corresponding fitting curves

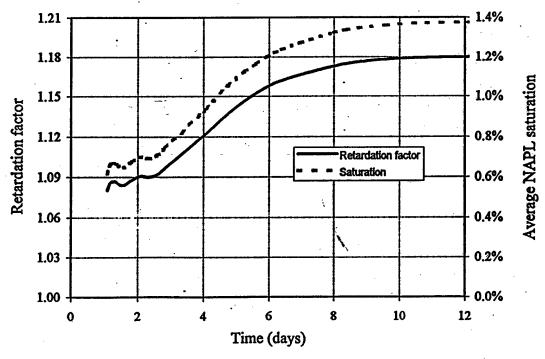


Figure 5b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS11\_BLUE

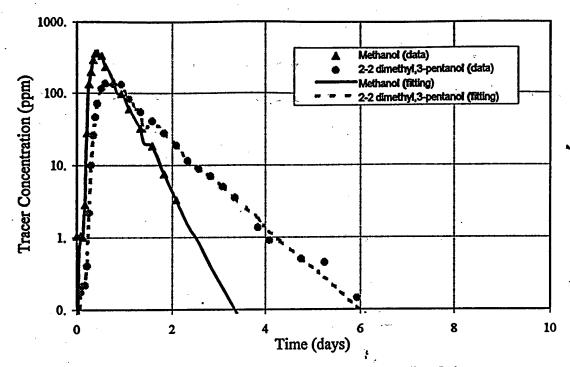


Figure 6a MLS11\_RED tracer response data and the corresponding fitting curves

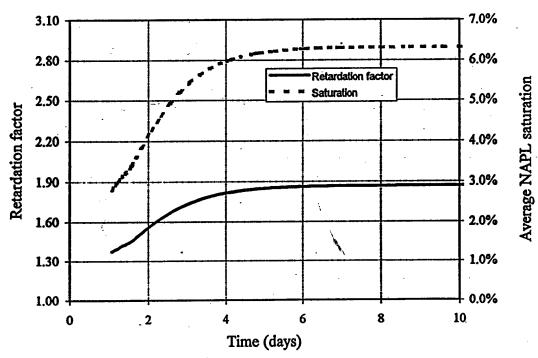


Figure 6b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS11\_RED

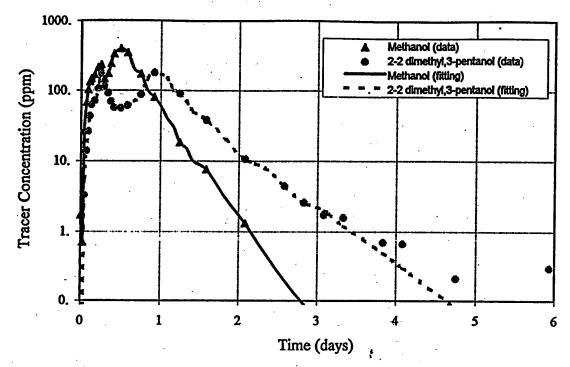


Figure 7a MLS11\_WHITE tracer response data and the corresponding fitting curves

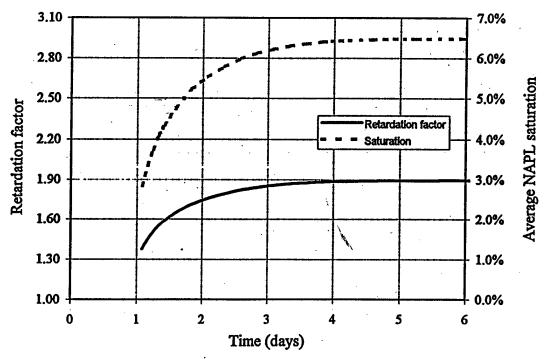


Figure 7b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS11\_WHITE

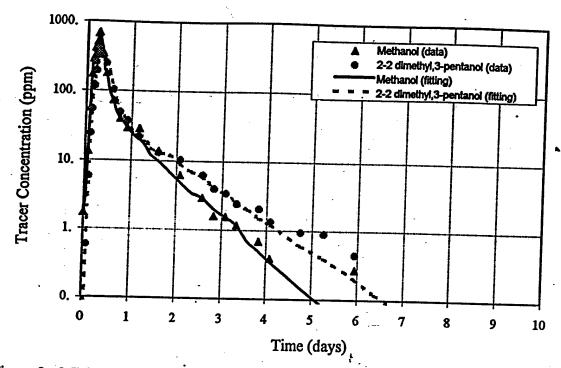


Figure 8a MLS11\_YELLOW tracer response data and the corresponding fitting curves

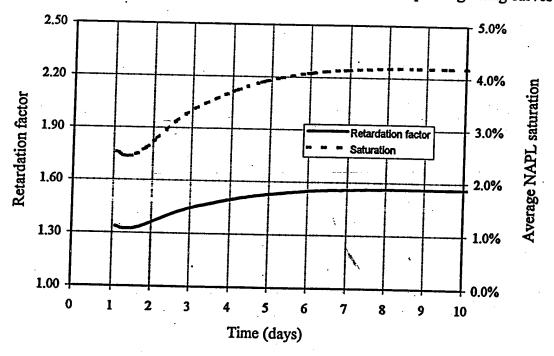


Figure 8b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS11\_YELLOW

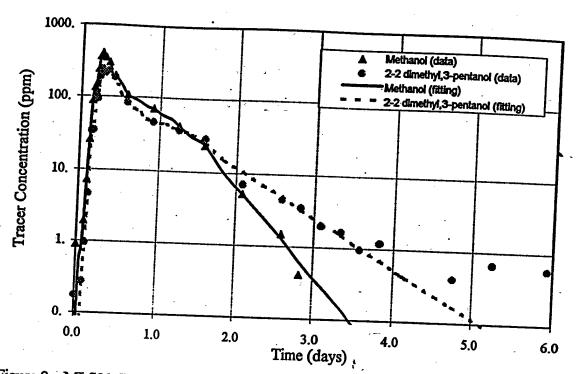


Figure 9a MLS21\_BLACK tracer response data and the corresponding fitting curves

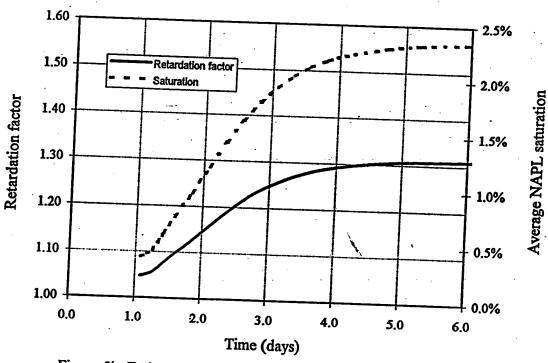


Figure 9b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS21\_BLACK

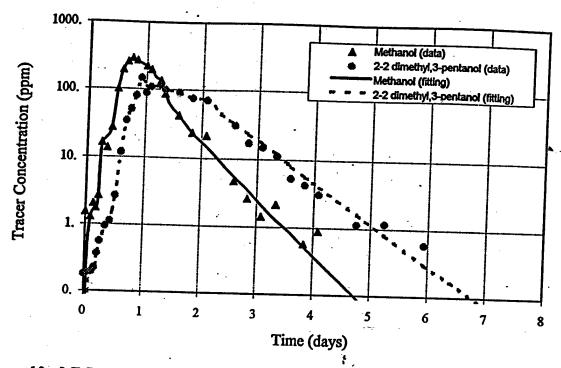


Figure 10a MLS21\_BLUE tracer response data and the corresponding fitting curves

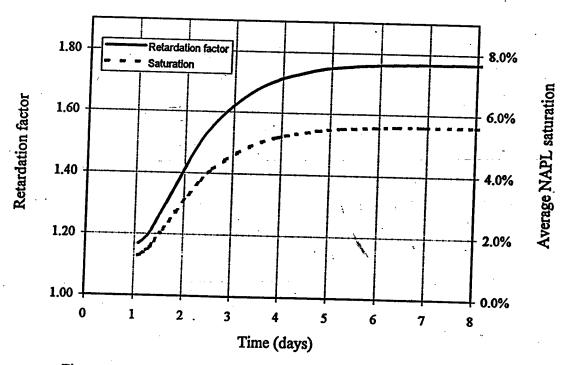


Figure 10b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS21\_BLUE

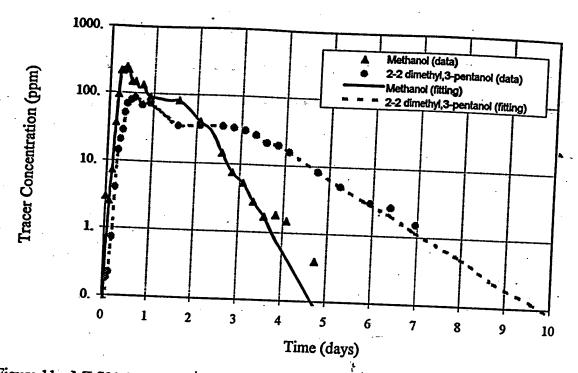


Figure 11a MLS21\_RED tracer response data and the corresponding fitting curves

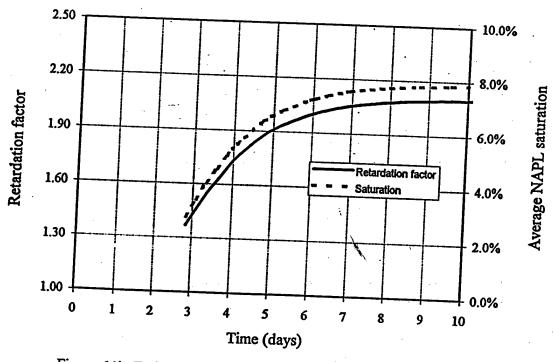


Figure 11b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS21\_RED

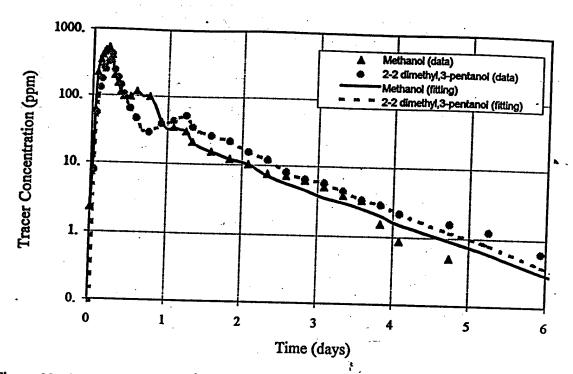


Figure 12a MLS21\_WHITE tracer response data and the corresponding fitting curves

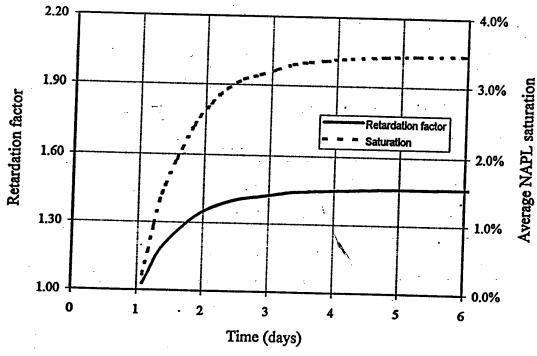


Figure 12b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS21\_WHITE

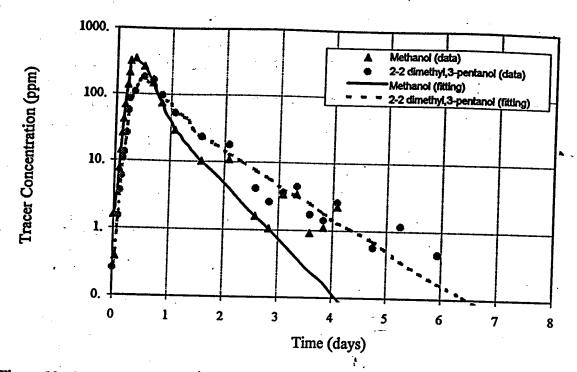


Figure 13a MLS21\_YELLOW tracer response data and the corresponding fitting curves

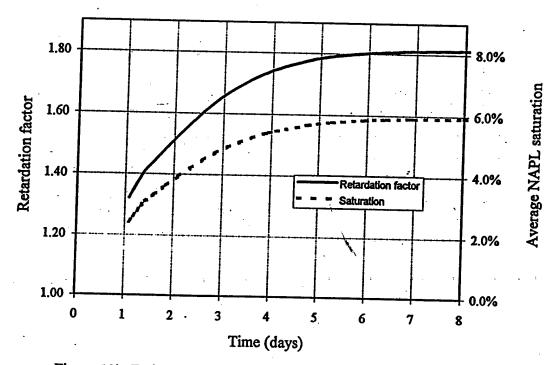


Figure 13b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS21\_YELLOW

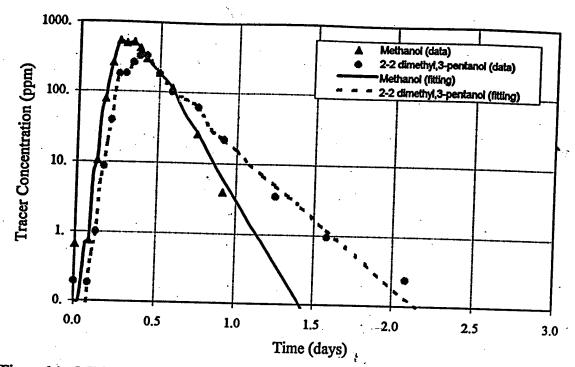


Figure 14a MLS31\_BLACK tracer response data and the corresponding fitting curves

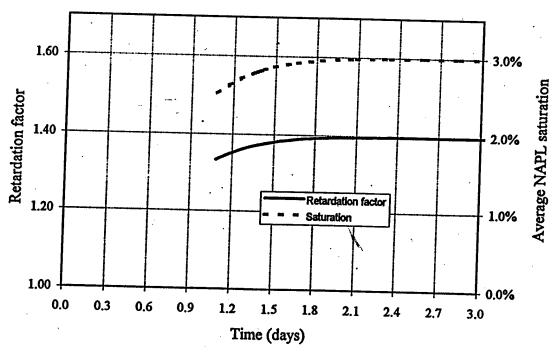


Figure 14b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS31\_BLACK

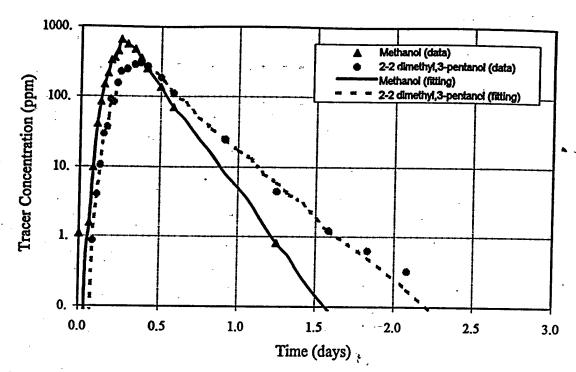


Figure 15a MLS31\_BLUE tracer response data and the corresponding fitting curves

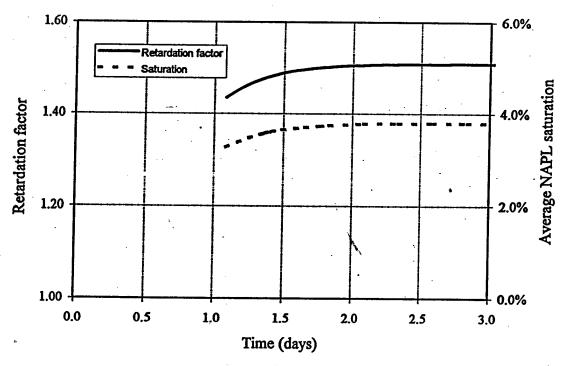


Figure 15b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS31\_BLUE

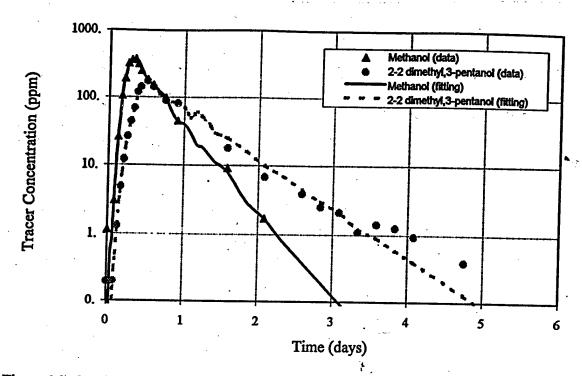


Figure 16a MLS31\_RED tracer response data and the corresponding fitting curves

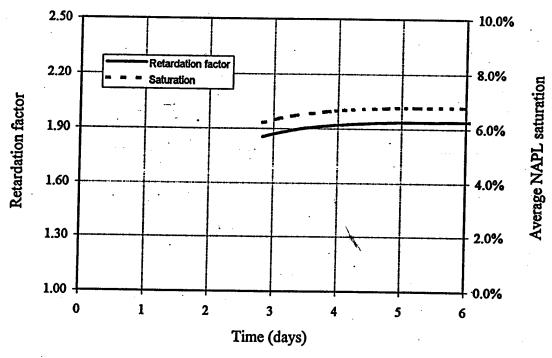


Figure 16b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS31\_RED

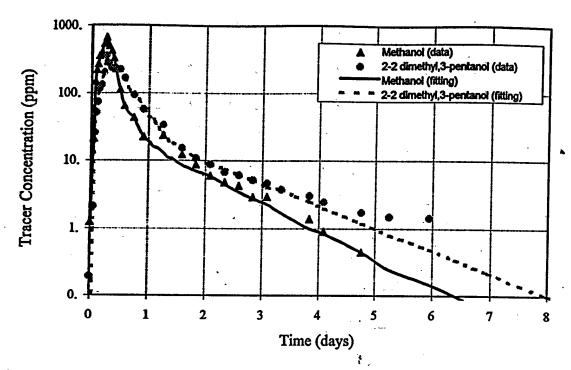


Figure 17a MLS31\_WHITE tracer response data and the corresponding fitting curves

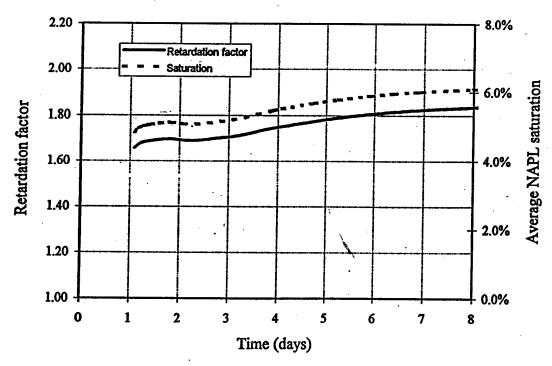


Figure 176 Estimated retardation factor and residual NAPL saturation based on tracer data at MLS31\_WHITE

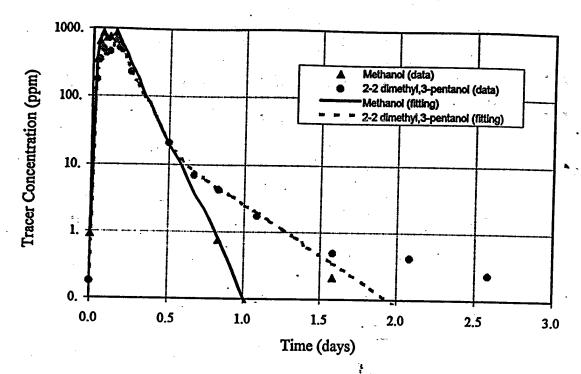


Figure 18a MLS31\_YELLOW tracer response data and the corresponding fitting curves

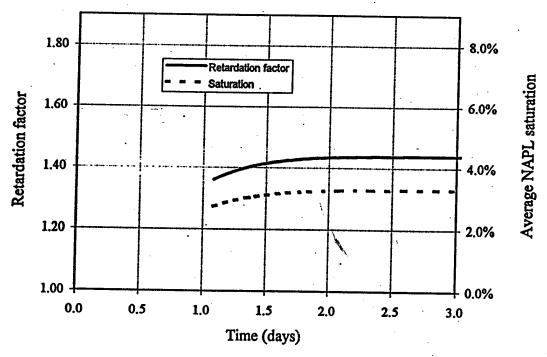


Figure 18b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS31\_YELLOW

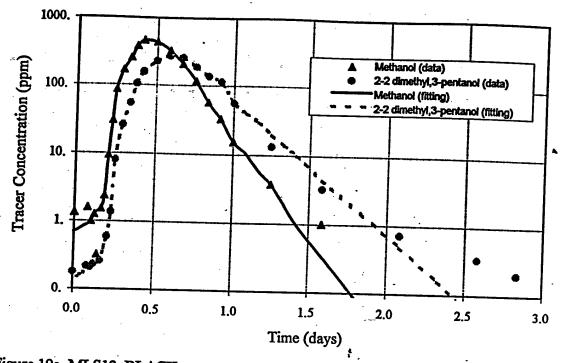


Figure 19a MLS12\_BLACK tracer response data and the corresponding fitting curves

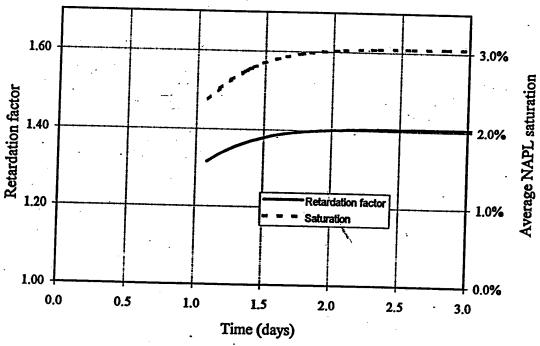


Figure 19b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS12\_BLACK

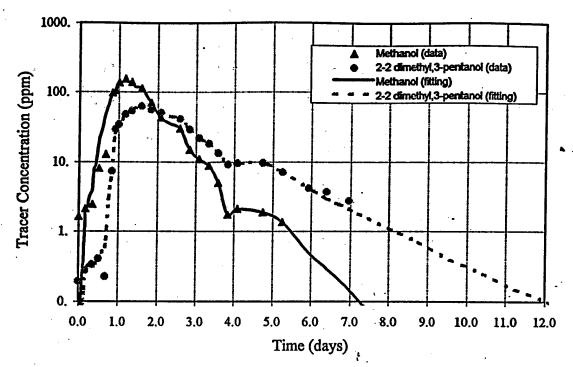


Figure 20a MLS12\_BLUE tracer response data and the corresponding fitting curves

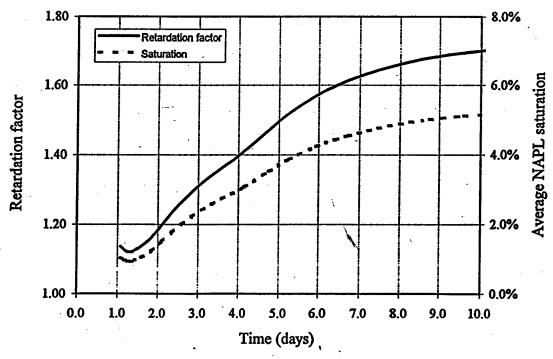


Figure 20b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS12\_BLUE

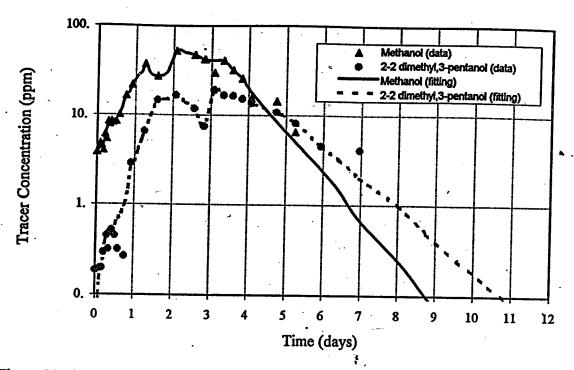


Figure 21a MLS12\_RED tracer response data and the corresponding fitting curves

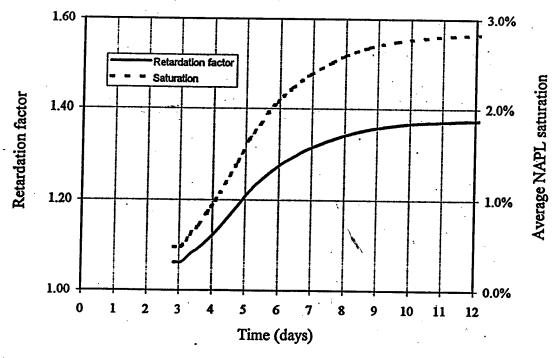


Figure 21b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS12\_RED

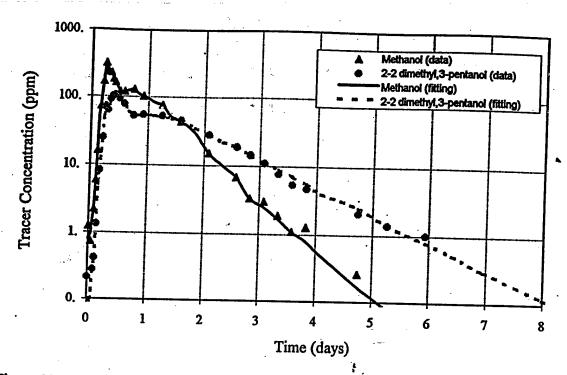


Figure 22a MLS12\_WHITE tracer response data and the corresponding fitting curves

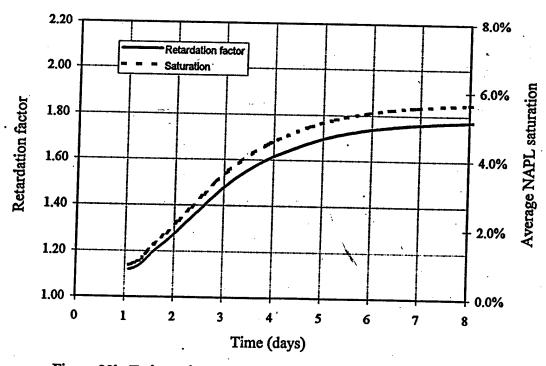


Figure 22b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS12\_WHITE

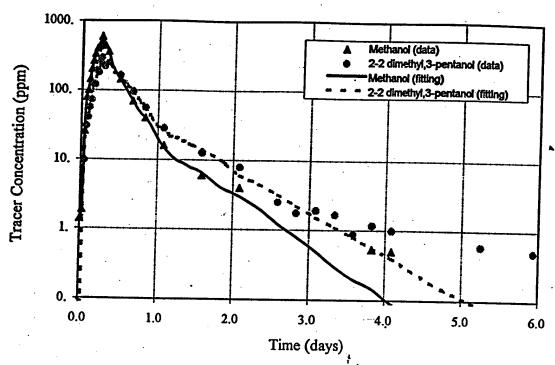


Figure 23a MLS12\_YELLOW tracer response data and the corresponding fitting curves

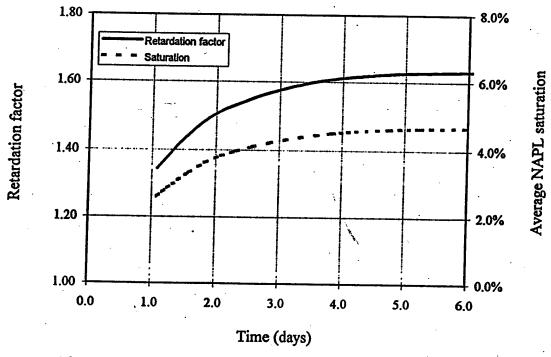


Figure 23b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS12\_YELLOW

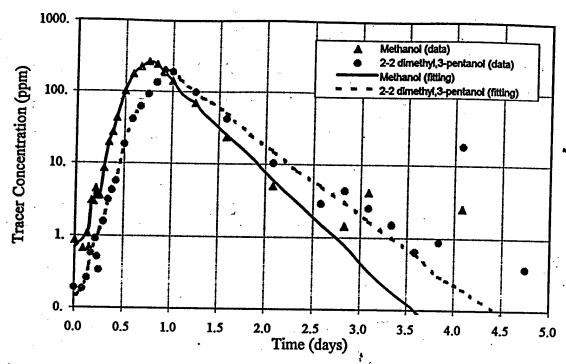


Figure 24a MLS22\_BLACK tracer response data and the corresponding fitting curves

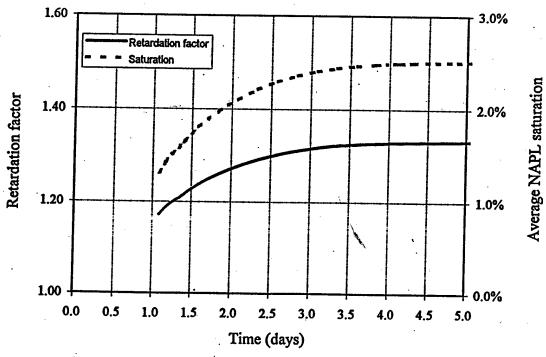


Figure 24b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS22\_BLACK

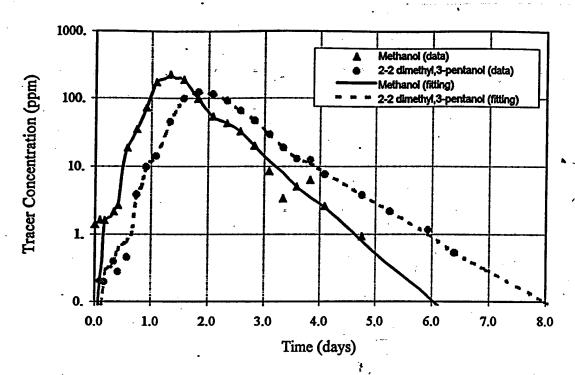


Figure 25a MLS22\_BLUE tracer response data and the corresponding fitting curves

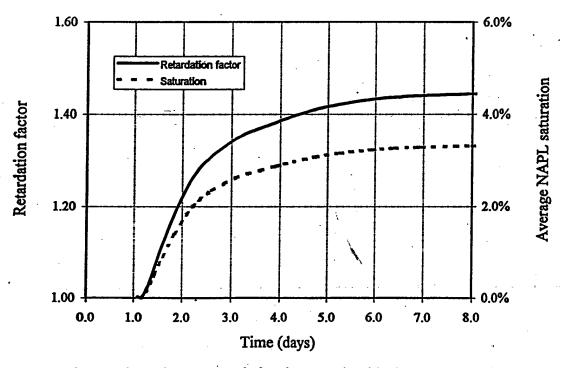


Figure 25b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS22\_BLUE

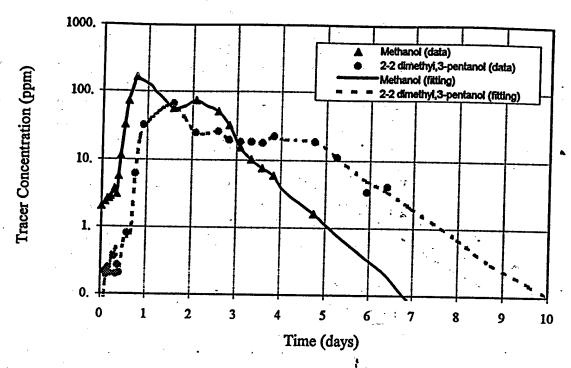


Figure 26a MLS22\_RED tracer response data and the corresponding fitting curves

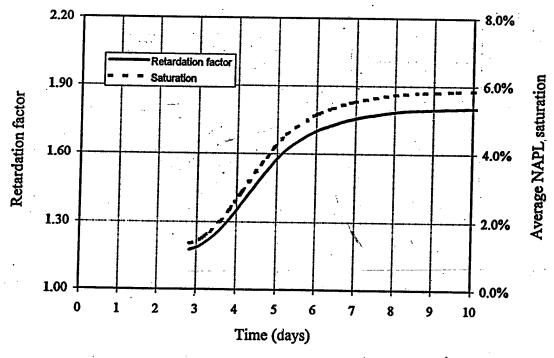


Figure 26b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS22\_RED

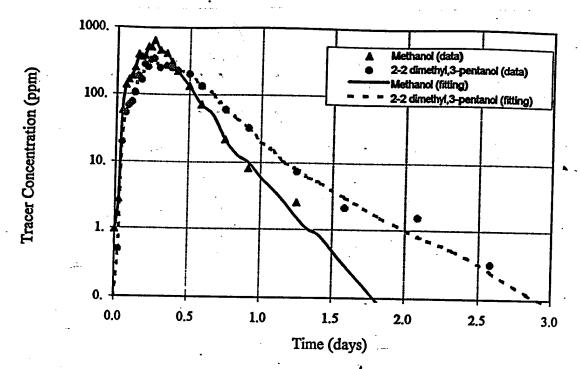


Figure 28a MLS22\_YELLOW tracer response data and the corresponding fitting curves

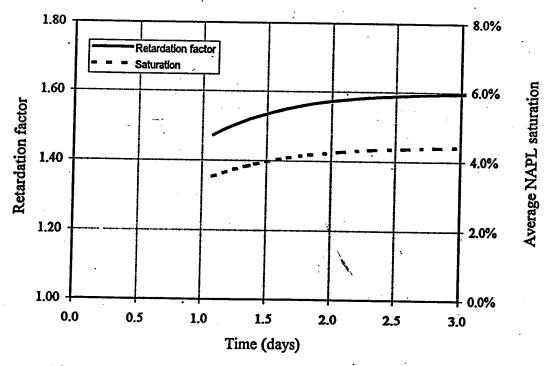


Figure 28b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS22\_YELLOW

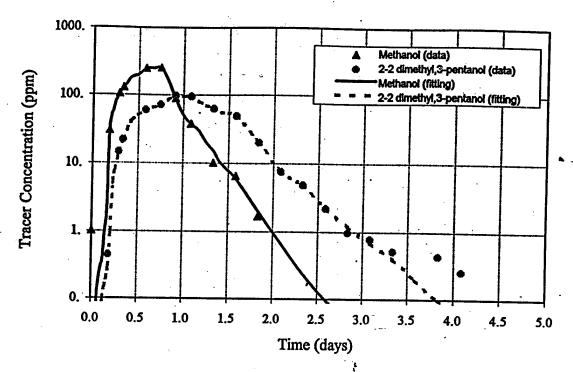


Figure 29a MLS32\_BLUE tracer response data and the corresponding fitting curves

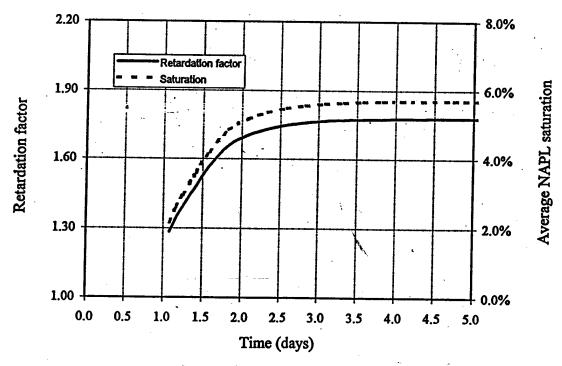


Figure 29b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS32\_BLUE

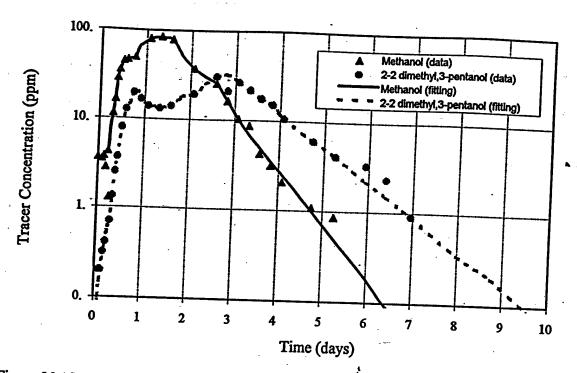


Figure 30a MLS32\_RED tracer response data and the corresponding fitting curves

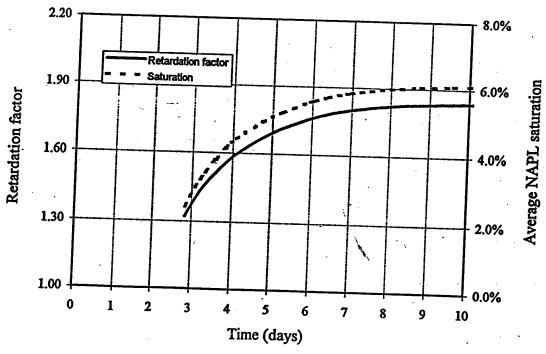


Figure 30b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS32\_RED

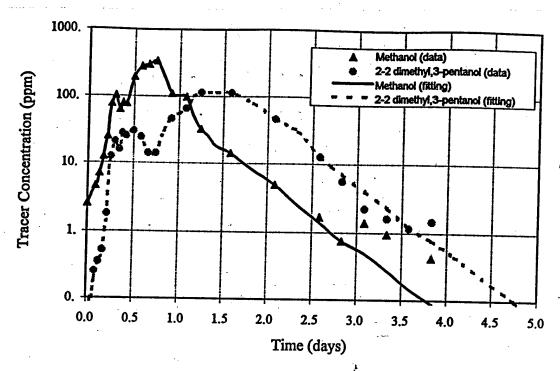


Figure 31a MLS32\_WHITE tracer response data and the corresponding fitting curves

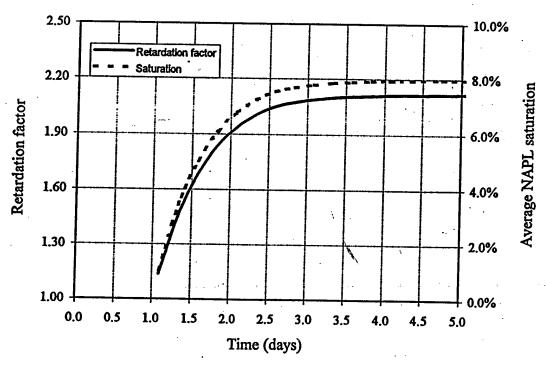


Figure 31b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS32\_WHITE

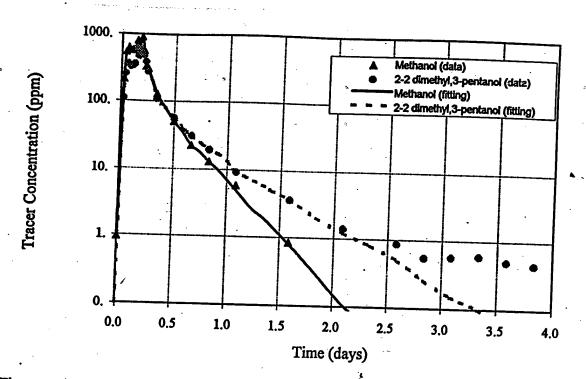


Figure 32a MLS32\_YELLOW tracer response data and the corresponding fitting curves

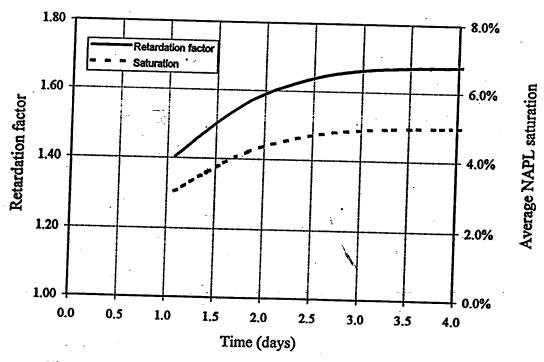


Figure 32b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS32\_YELLOW

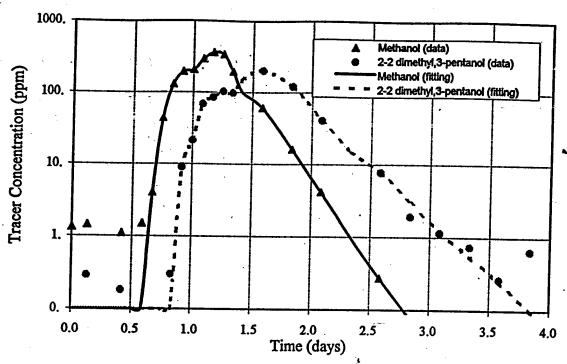


Figure 33a MLS13\_BLACK tracer response data and the corresponding fitting curves

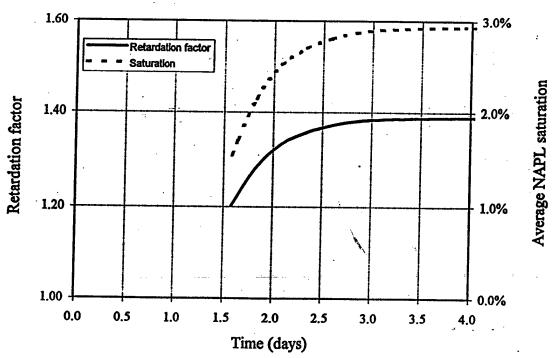


Figure 33b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS13\_BLACK

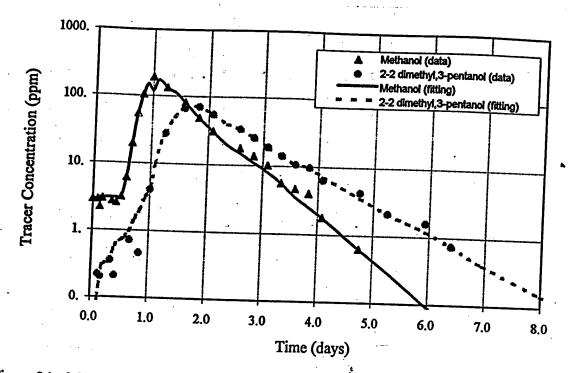


Figure 34a MLS13\_BLUE tracer response data and the corresponding fitting curves

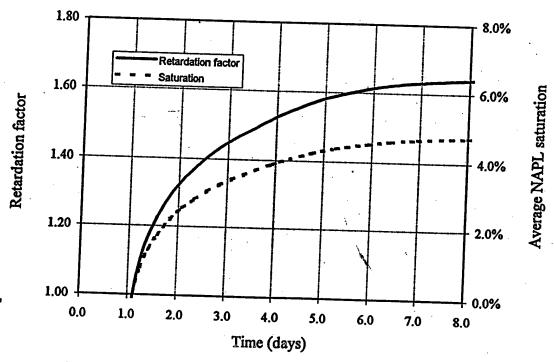


Figure 34b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS13\_BLUE

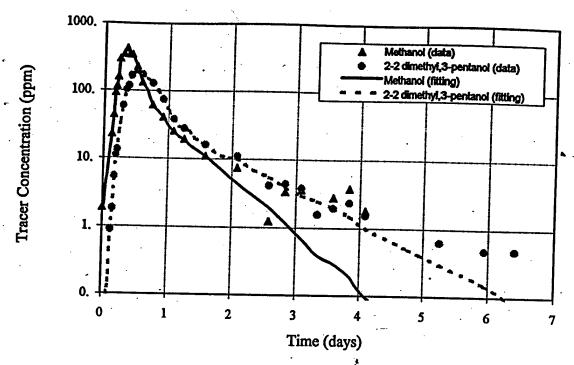


Figure 35a MLS13\_WHITE tracer response data and the corresponding fitting curves

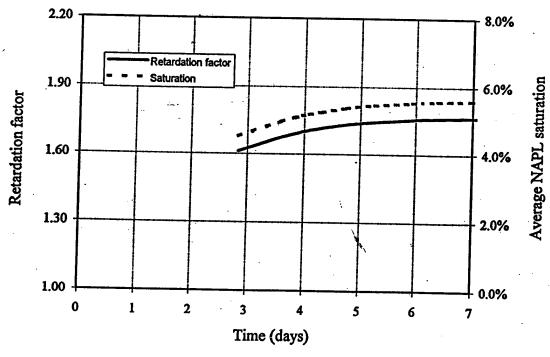


Figure 35b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS13\_WHITE

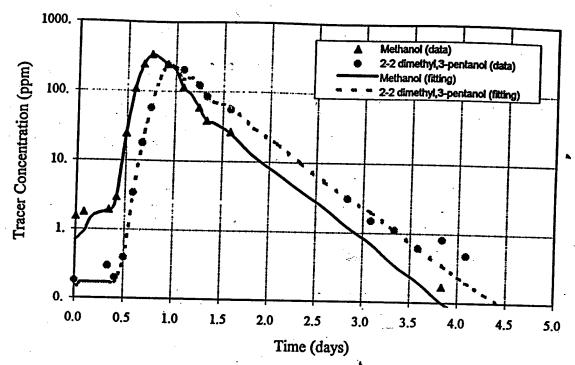


Figure 36a MLS23\_BLACK tracer response data and the corresponding fitting curves

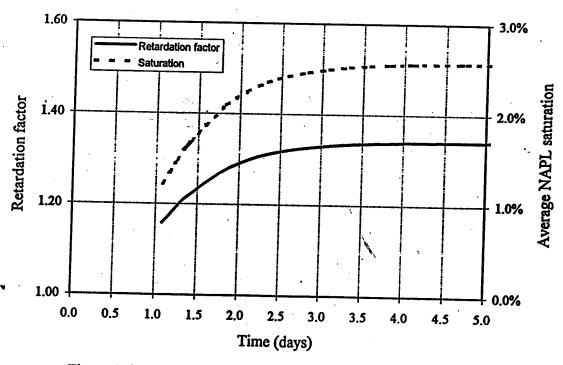


Figure 36b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS23\_BLACK

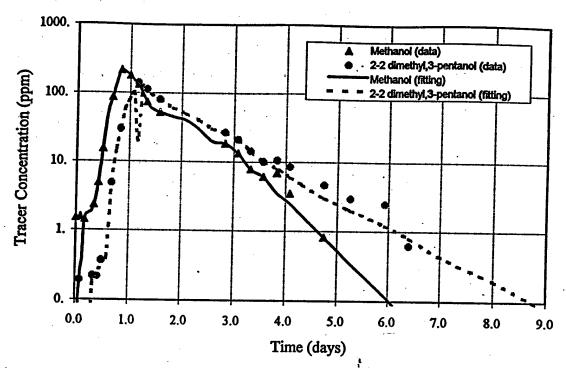


Figure 37a MLS23\_BLUE tracer response data and the corresponding fitting curves

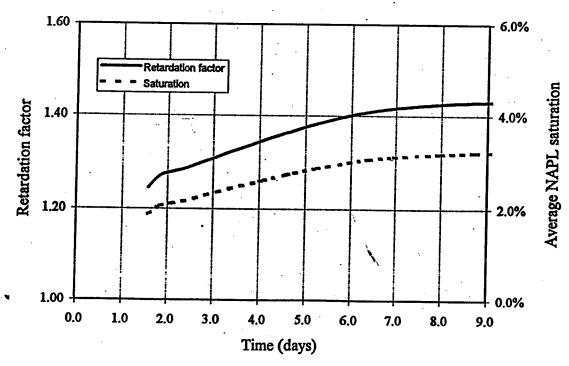


Figure 37b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS23\_BLUE

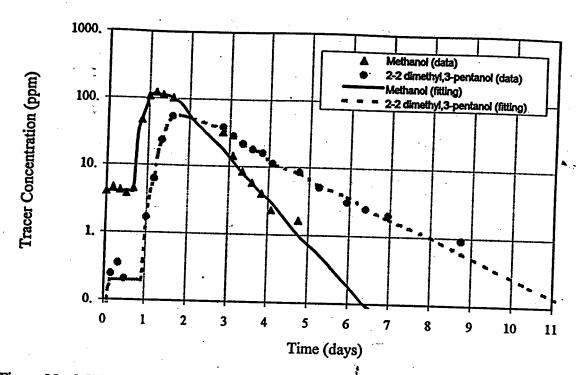


Figure 38a MLS23\_RED tracer response data and the corresponding fitting curves

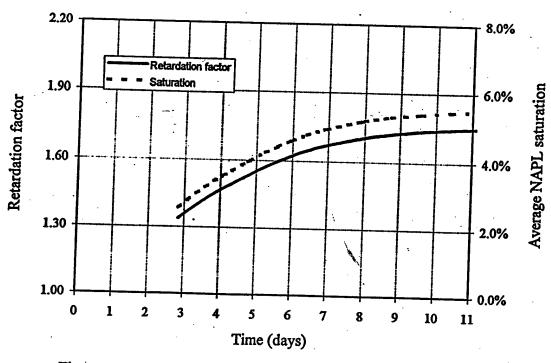


Figure 38b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS23\_RED

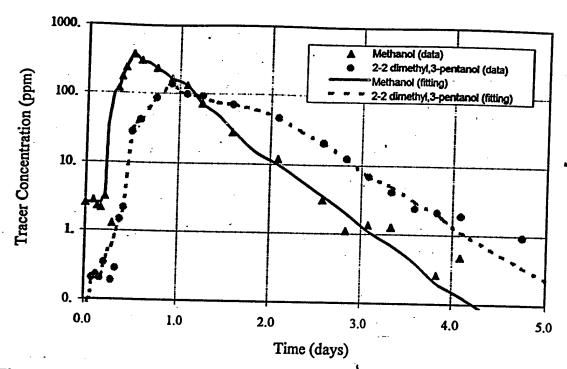


Figure 39a MLS23\_WHITE tracer response data and the corresponding fitting curves

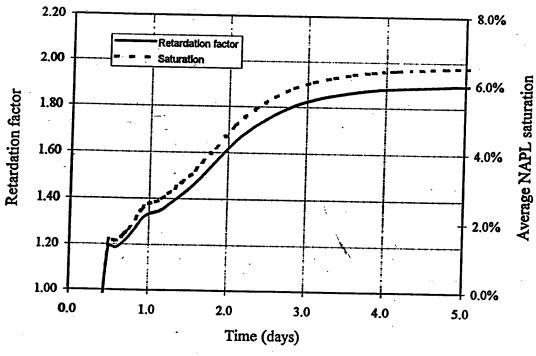


Figure 39b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS23\_WHITE

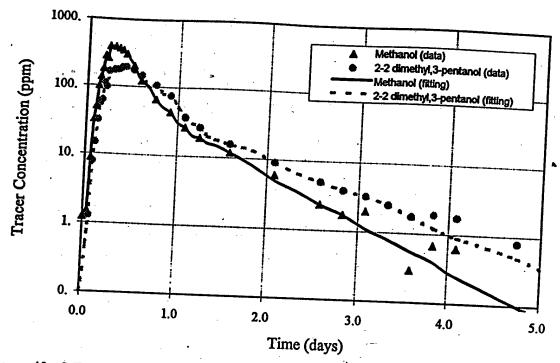


Figure 40a MLS23\_YELLOW tracer response data and the corresponding fitting curves

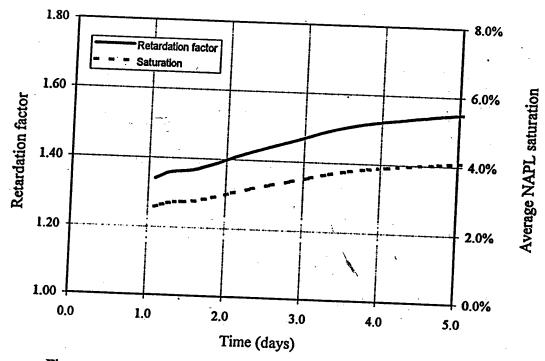


Figure 40b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS23\_YELLOW

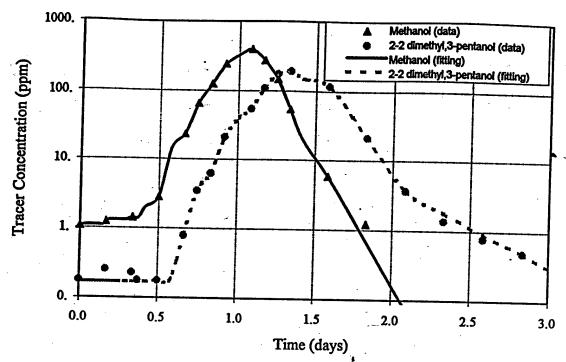


Figure 41a MLS33\_BLACK tracer response data and the corresponding fitting curves

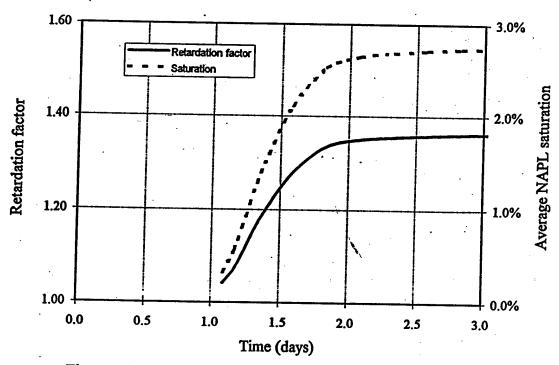


Figure 41b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS33\_BLACK

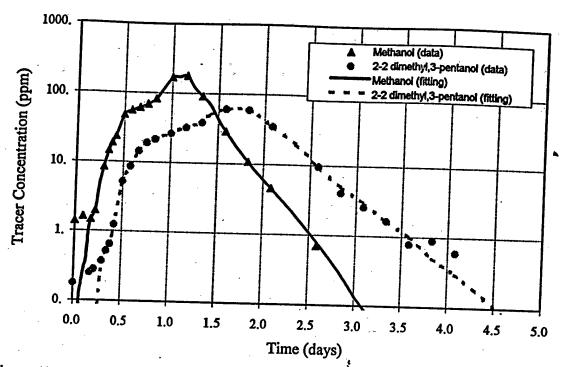


Figure 42a MLS33\_BLUE tracer response data and the corresponding fitting curves

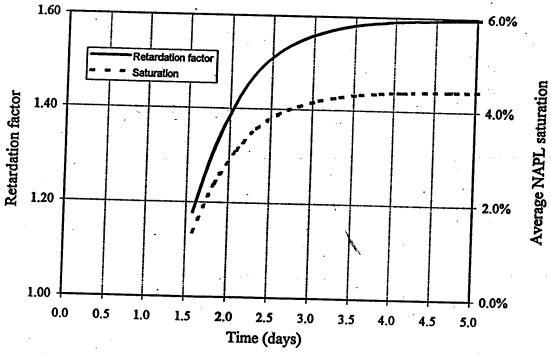


Figure 42b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS33\_BLUE

Figure 3.7 Shear wave time history for location H-LWF-C04 70 to 150 ft.

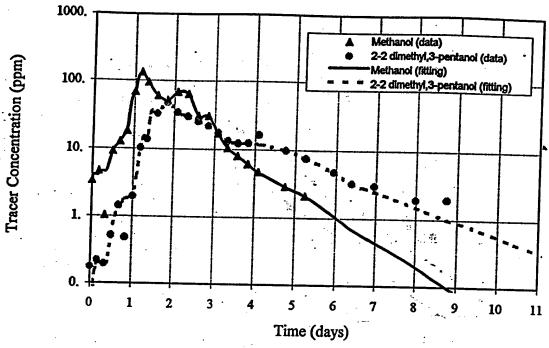


Figure 43a MLS33\_RED tracer response data and the corresponding fitting curves

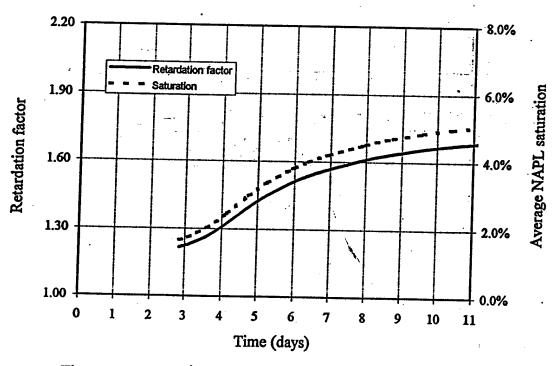


Figure 43b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS33\_RED

Figure 3.8 Shear wave time history for location H-CWF-CO4 140 to 157.7 ft.

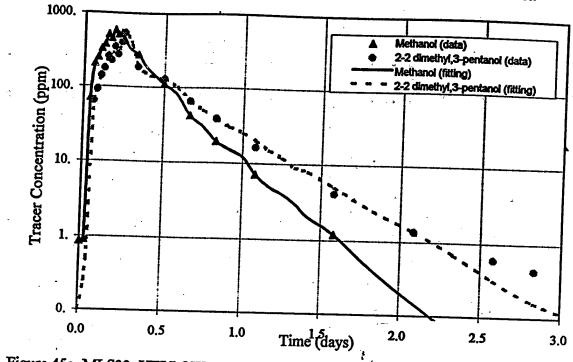


Figure 45a MLS33\_YELLOW tracer response data and the corresponding fitting curves

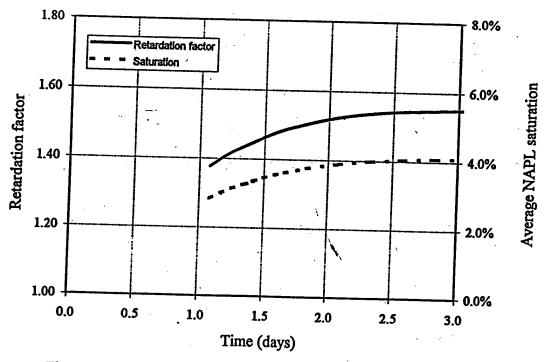


Figure 45b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS33\_YELLOW

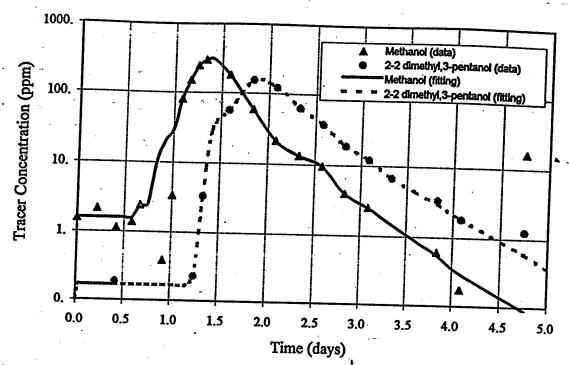


Figure 46a MLS14\_BLACK tracer response data and the corresponding fitting curves

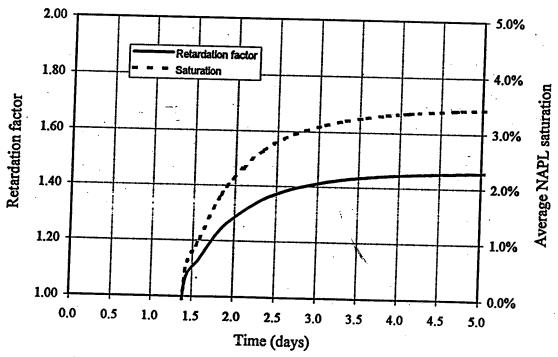


Figure 46b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS14\_BLACK

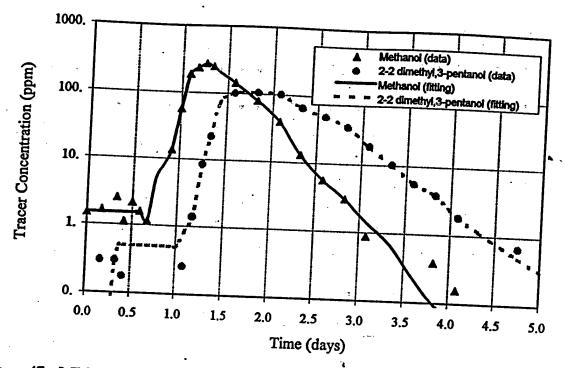


Figure 47a MLS14\_BLUE tracer response data and the corresponding fitting curves

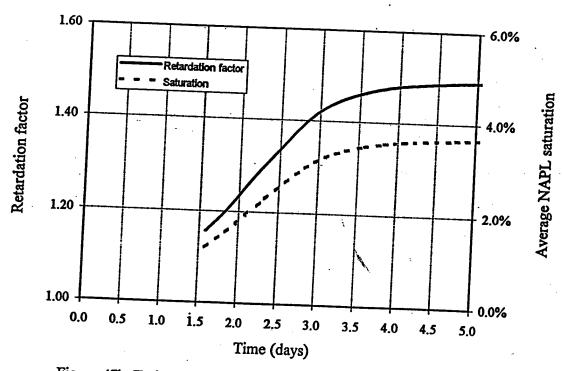


Figure 47b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS14\_BLUE

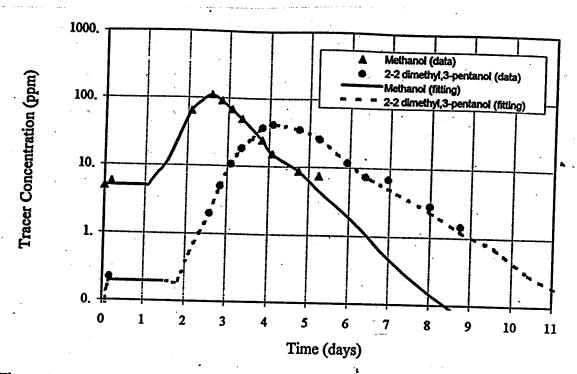


Figure 48a MLS14\_RED tracer response data and the corresponding fitting curves

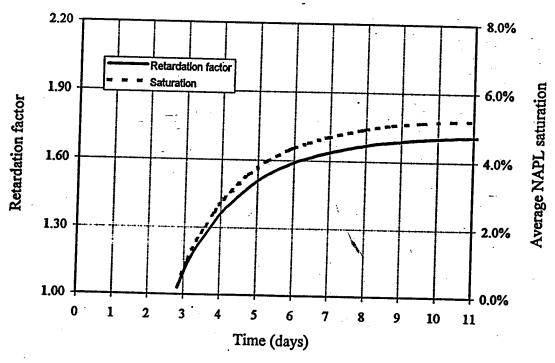


Figure 48b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS14\_RED

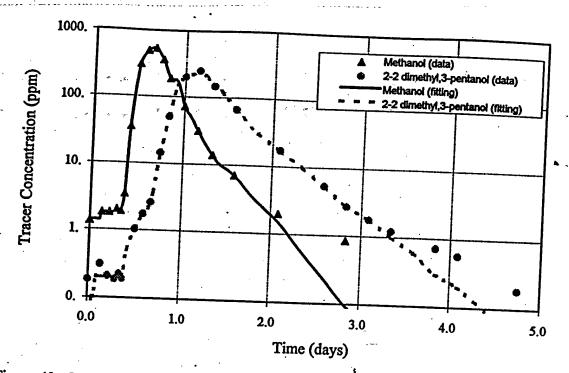


Figure 49a MLS14\_WHITE tracer response data and the corresponding fitting curves

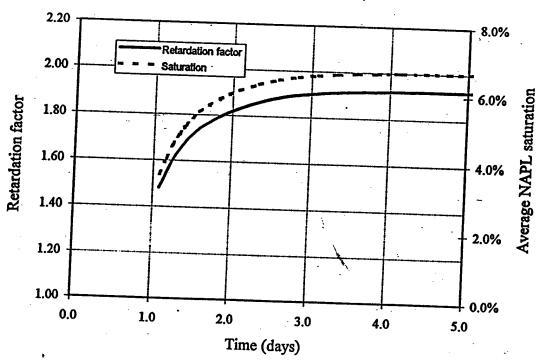


Figure 49b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS14\_WHITE

Figure 4.1 Compression wave time to peak for location H-LWF-C04 70 to 150 ft.

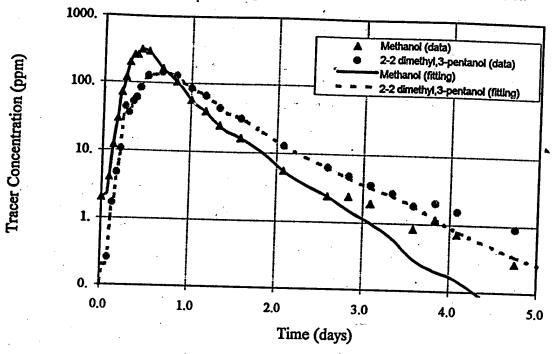


Figure 50a MLS14\_YELLOW tracer response data and the corresponding fitting curves

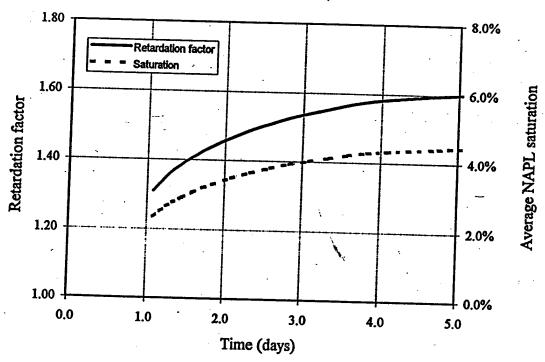


Figure 50b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS14\_YELLOW

. Figure 4.2 Compressional wave velocity peak for location H-CIF-C08.

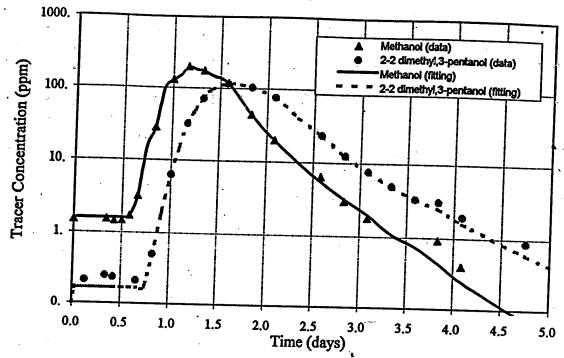


Figure 51a MLS24\_BLACK tracer response data and the corresponding fitting curves

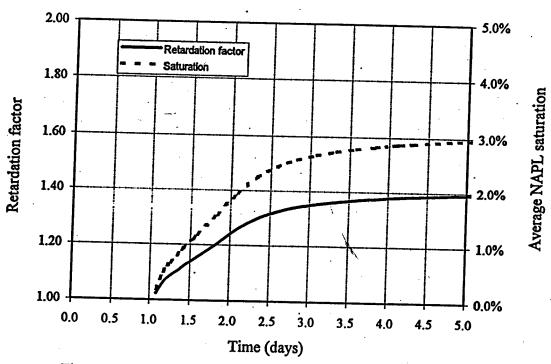


Figure 51b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS24\_BLACK

Figure 4.3 Shear wave time to peak for location H-CIF-C10.

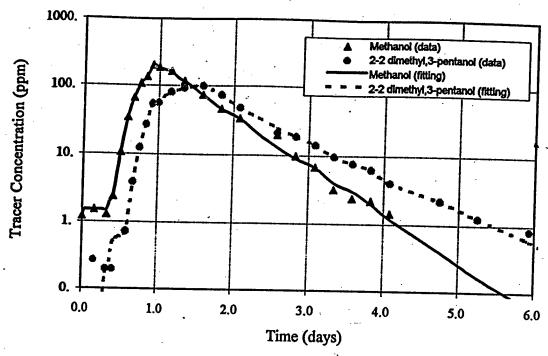


Figure 52a MLS24\_BLUE tracer response data and the corresponding fitting curves

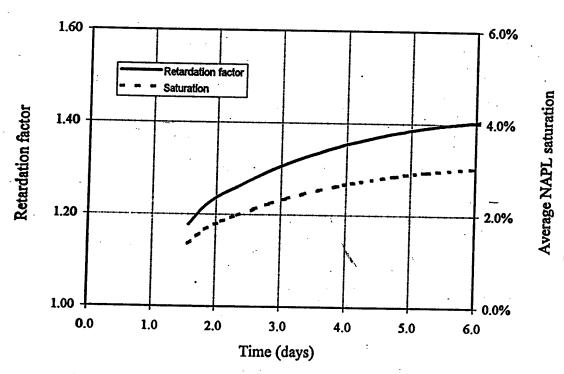


Figure 52b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS24\_BLUE

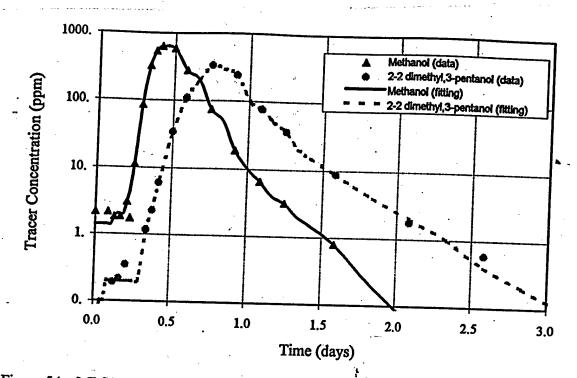


Figure 54a MLS24\_WHITE tracer response data and the corresponding fitting curves

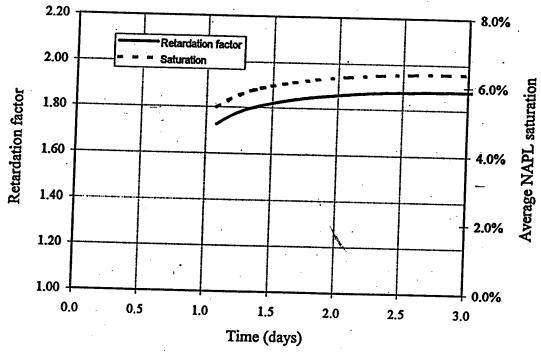


Figure 54b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS24\_WHITE



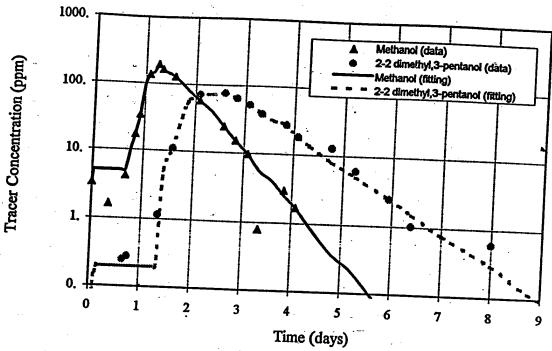


Figure 53a MLS24\_RED tracer response data and the corresponding fitting curves

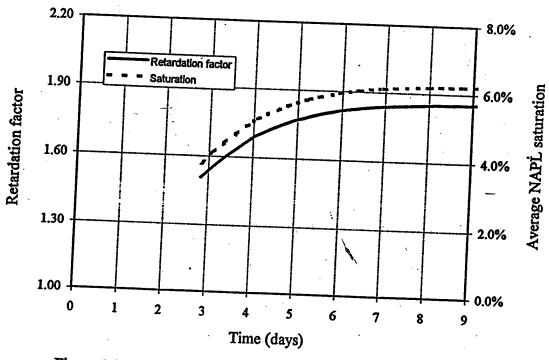


Figure 53b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS24\_RED

Figure 4.6 Average shear wave particle velocity location H-CIF-C10.

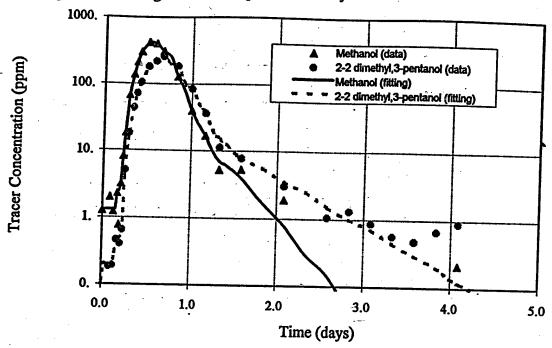


Figure 55a MLS24\_YELLOW tracer response data and the corresponding fitting curves

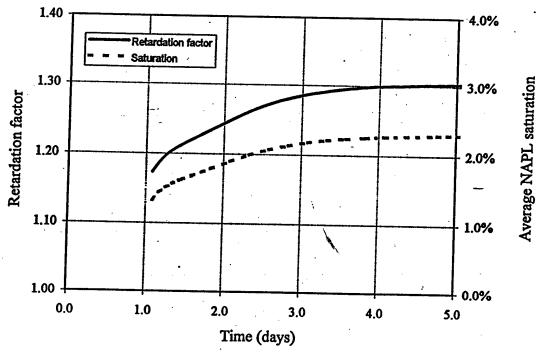


Figure 55b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS24\_YELLOW

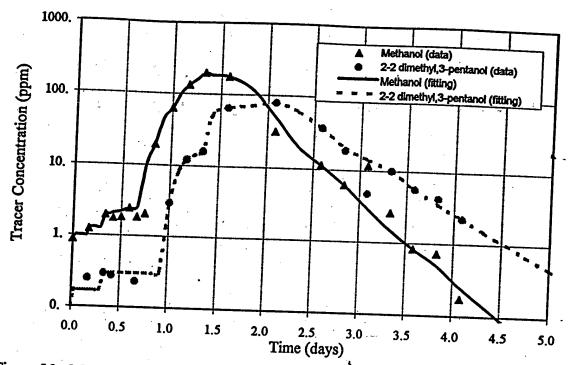


Figure 56a MLS34\_BLACK tracer response data and the corresponding fitting curves

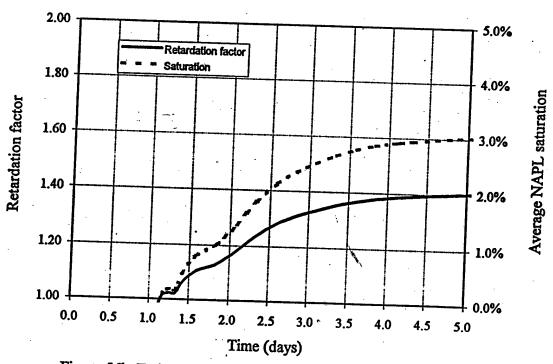


Figure 56b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS34\_BLACK

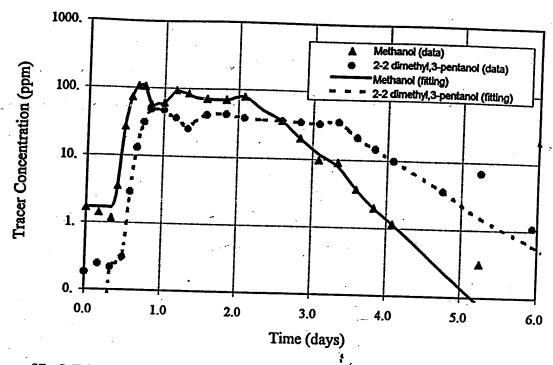


Figure 57a MLS34\_BLUE tracer response data and the corresponding fitting curves

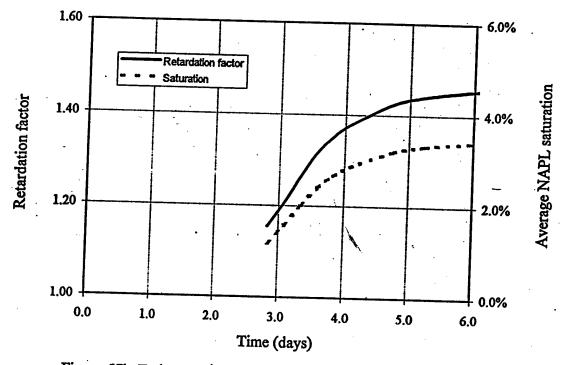


Figure 57b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS34\_BLUE

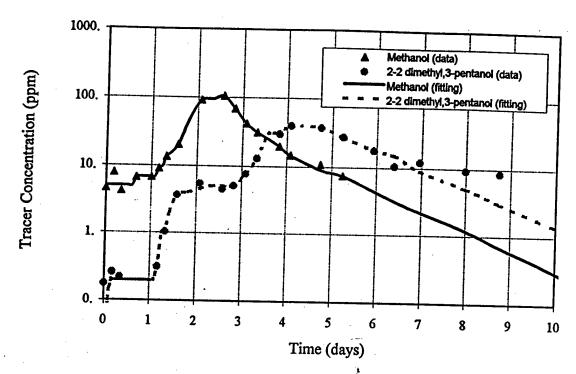


Figure 58a MLS34\_RED tracer response data and the corresponding fitting curves

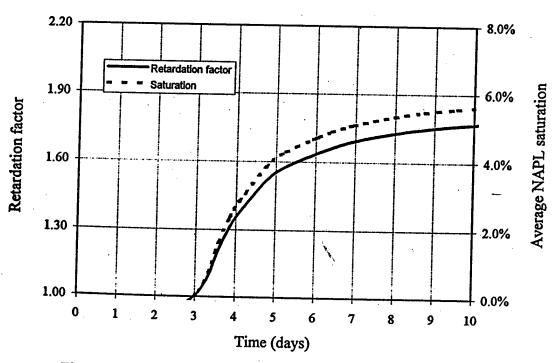


Figure 58b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS34\_RED

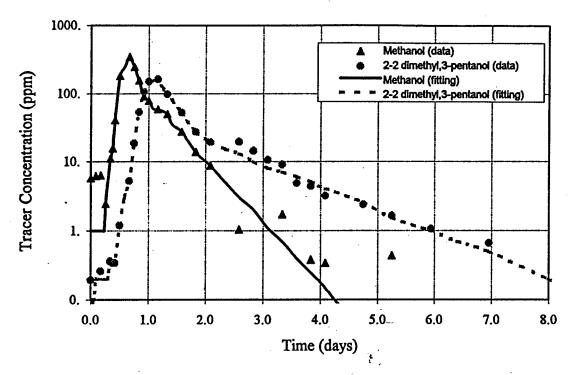


Figure 59a MLS34\_WHITE tracer response data and the corresponding fitting curves

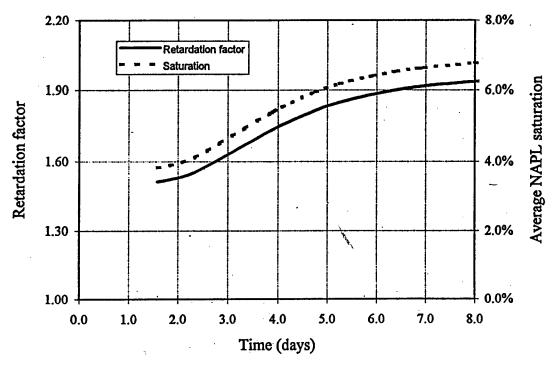


Figure 59b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS34\_WHITE

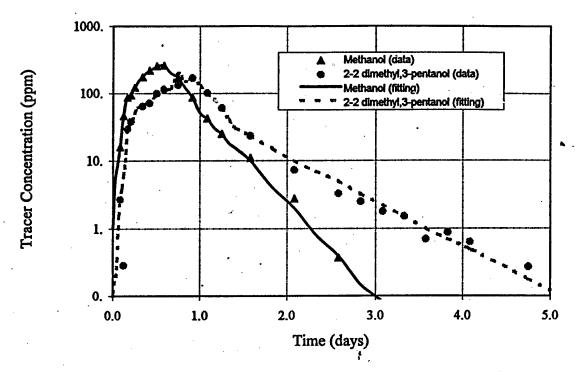


Figure 60a MLS34\_YELLOW tracer response data and the corresponding fitting curves

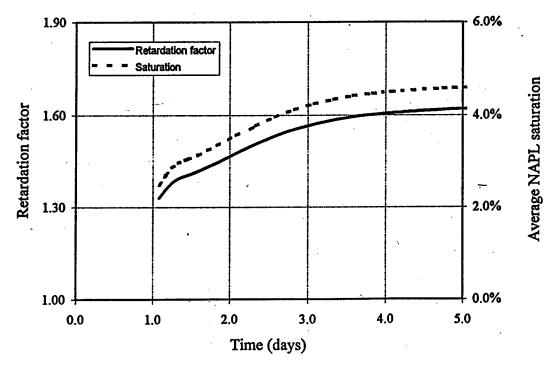


Figure 60b Estimated retardation factor and residual NAPL saturation based on tracer data at MLS34\_YELLOW

#### APPENDIX F

#### RSKSOP-148

STANDARD OPERATING PROCEDURE
DETERMINATION OF VOLATILE ORGANIC COMPOUNDS
IN WATER BY AUTOMATED HEADSPACE
GAS CHROMATOGRAPHY/MASS SPECTOMETERY
(SATURN II ION TRAP DETECTOR)



Ref: 95/JAD45

September 1, 1995

Mr. Lynn Wood

R.S. Kerr Environmental Research Lab

U.S. Environmental Protection Agency

P.O. Box 1198

Ada, OK 74820

THRU: S.A. Vandegrift  $6^{\checkmark}$ 

Dear Lynn:

As requested in Service Request # SF-1-130, headspace GC/MS analysis of Hill AFB water samples for VOC's was completed for the specified compounds: 1,1,1-trichloroethane, toluene, 1,2,-dichlorobenzene, 1,3,5-trimethylbenzene, o-xylene, m+p-xylene, decane, and naphthalene. A total of 60 water samples and 12 duplicates were received, in 20 ml VOA vials, on July 20, 1995. Samples were analyzed on August 6-12, 1995. RSKSOP-148 (Determination of Volatile Organic Compounds in Water by Automated Headspace Gas Chromatography/Mass Spectrometry (Saturn II Ion Trap Detector) was used for this analysis.

The following modifications were made to RSKSOP-148. Lead lined septum was used on the headspace vials. The filament delay was extended to 300 seconds. The acquisition time was extended to 25 minutes. Also the total run time was extended to 55 minutes.

An internal standard calibration method was established for the compounds. The decane curve ranged from 2.0 to 200 ppb. 1,2-Dichlorobenzene ranged from 2.0 to 4000 ppb. Naphthalene ranged from 5.0 to 2000. The other compounds ranged from 1.0 to 2000 ppb. The internal standard was fluorobenzene at a concentration of 100 ppb in the headspace vial.

A quantitation report for the samples, lab duplicates, field duplicates, QC standards and lab blanks is presented in Table 1.

Note that because of a computer acquisition failure, samples sets M8 thru M12 were rerun. The second 10 ml aliquot was taken from the 20 ml VOA vials. A 10 ml volume of headspace was above 10 ml sample for 48 hours. The field duplicates without headspace were run to compare possible losses.

Evaluation of the data reveals that duplicate analyses of the second aliquots of sample from the 20 ml VOA vials had higher

ManTech Environmental Research Services Corporation

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### STANDARD OPERATING PROCEDURE

DETERMINATION OF VOLATILE ORGANIC COMPOUNDS IN WATER BY AUTOMATED HEADSPACE GAS CHROMATOGRAPHY/MASS SPECTROMETRY (SATURN II ION TRAP DETECTOR)

<u>Disclaimer:</u> This Standard Operating Procedure has been prepared for the use of the Robert S. Kerr Environmental Research Laboratory of the U.S.E.P.A. and may not be specifically applicable to the activities of other organizations.

I. <u>Purpose</u>: (Scope and Application)

This method is applicable to the confirmed identification and quantitation of purgeable volatile organic compounds (VOC) in water using gas chromatography/mass spectrometry (GC/MS) without the use of cryogenic focusing. It is based on EPA Method 524.2 which is used to determine over sixty VOC in drinking water. In addition to the analysis of aromatics and haloalkenes, haloalkanes and haloaromatics, this method can also be used for the determination of petroleum hydrocarbons in contaminated water. The method is automated in that sampling and GC/MS analysis occur automatically after minimum input from the operator.

Approximately eleven analytical runs can be performed per eight hour day. The sample tray can be loaded with 50 samples which can be analyzed within thirty-eight hours.

This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography/mass spectrometry and in the interpretation of chromatograms and mass spectra.

### II. Summary of Method:

A sample is sealed in a headspace vial and heated in the sampler's aluminum platen. The headspace gas in the vial is then sampled with a needle, flushed via a transfer line into a capillary column at 35°C with no cryogenic cooling in the GC oven which provides compound separation. The volatile organics are identified and quantified by the GC/MS software system. Based on a 10 ml sample

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size a sample concentration in the range of 0.5 - 1,000 ppb can be determined. A method detection limit for 21 aromatic, haloalkane, haloalkene and haloaromatic compounds was determined at a concentration of 0.5 ppb and ranged from 0.06 to 0.5 ppb (See Table III).

#### III. References:

- 1. Methods for Determination of Organic Compounds in Drinking Water Method 524.2 Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry. EPA/600/4-98/039, Dec. 1988.
- 2. Tekmar 7000 Headspace Sampler Operating and Service Manual, Tekmar Co., Cincinnati, OH.
- 3. Varian 3300/3400 Gas Chromatograph Operators Manual, Varian Analytical Instruments, Sugar Land, TX.
- 4. Saturn II GC/MS Operatoring/Reference/Applications Software Manuals, Varian Analytical Instruments, Sugar Land, TX.
- 5. Eichelberger, J.W., Bellar, T.A., Donnelly, J.P. and Budde, W.L., Determination of Volatile Organics in Drinking Water with USEPA Method 524.2 and the Ion Trap Detector. Journal of Chromatographic Science, Vol. 28, pp.460-467, Sept. 1990.
- 6. Definition and Procedure for the Determination of the Method Detection Limit Revision 1.11, Code of Federal Regulations, 40CFR Ch.I (7-1-91 Edition), Pt. 136, App. B, Environmental Protection Agency, pp. 554 555.
- 7. Environmental Chromatography Seminar 1992, Varian Analytical Instruments, San Fernando, CA.
- 8. RSKSOP-71 Revision No. 4, Date: 11/14/91, Steve Vandegrift, RSKERL, Ada, OK.
- 9. RSKSOP-127 Revision No. 1, Date: 05/28/92, Dennis Fine, RSKERL, ADA, OK.

#### IV. Procedure:

#### A. Sample Preparation

Weigh out 2.0 grams of sodium chloride into a 20 milliliter headspace vial. Using a 10 milliliter syringe, add 10 mls of

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sample, or in the case of a blank or standard preparation, organic free water.

- Note 1: For the analysis of a soil, weigh out 1-2 grams and record the weight, and then add 10 mls of water.

  Analytical results will be qualitative and semi-quantitative under these conditions.
- Note 2: Volatile organic-free water may be generated by boiling for one hour water that has undergone reverse osmosis and treatment by a Millipore water system.

Once the water or the sample has been added to the vial, quickly inject 8.0  $\mu$ l of fluorobenzene + bromofluorobenzene internal standard (See Note 3), under the surface of the water/sample. The vial should be quickly capped and crimped. When a standard is being prepared, a correct amount of standard solution should also be quickly injected under the surface of the water. It also should be rapidly capped and crimped.

After the sample or standard vial has been crimped, shake the vial to completely dissolve the salt.

Note 3: To prepare the internal standard, inject 6.1  $\mu$ l of neat fluorobenzene and 4.2  $\mu$ l of neat p-bromofluorobenzene into methanol in a 50 ml volumetric flask. Dilute to the mark with methanol. This results in concentrations of approximately 125 ng/ $\mu$ l.

Place the vials in the carousel in the order in which they are to be analyzed.

The analysis time for each sample will be approximately 35 minutes. This allows each sample to be heated at 80°C in the carousel of the headspace sampler for 30 minutes before it is analyzed.

### B. Readspace Sampling.

Check that the parameters used for operation of the headspace sampler are as listed in Section IV.

Enter the number of vials to be analyzed into the autosampler menu. (See the Tekmar manual for details.)

Be certain that the GC/MS is ready for operation by checking the GC/MS air/water ratio and operation paramaters (See section C).

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Press the start button on the headspace sampler and answer the GC cycle time question. The sampler will start automatically. After heating the sample, the headspace sampler begins its cycle by piercing the vial's septum with the sampling needle and pressurizing the vial with helium. The headspace gas is then vented through the one milliliter sample loop. The filled sample loop is placed in series with the GC column and its contents are transferred through the heated transfer line to the head of the

#### C. GC/XS.

Check that the parameters used for operation of the GC/MS are as listed in Section IV and in the GC/MS method files shown in Figures 1-2.

At the start of each analysis, the GC temperature program is downloaded to the GC by the GC/MS system software. Upon completion of analysis the GC will return to its initial set point of 35 °C and remain there.

The transfer line from the Tekmar 7000 is connected directly to the helium supply line of the split injection port of the Varian 3400 gas chromatograph. A constant split of 30 ml/min is maintained at the injector (with a column pressure of 12.5 psi, the air dead time at 80 °C of 83 sec is obtained through the analytical column). The interface between the GC and the MS is accomplished by extending the column through the transfer line. The column should extend 0.5 mm past the end of the transfer line. The transfer line is guided into the manifold until it will go no further. It is then clamped into place.

The Saturn II Ion Trap Detector (ITD) parameters are shown in Figures 3-4.

The criteria for tuning the ion trap detector is based on spectral parameters set by EPA Method 524.2 using p-bromofluorobenzene. These parameters are met before starting calibration of the system and are checked at the start of each day before beginning analysis. Figure 5 shows a spectrum and listing for a calibration check (generated by run procedure BFB524B) done on the Saturn II ITD system and the p-bromofluorobenzene tuning criteria.

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# IV. Instrument Operating Parameters:

### A. Headspace Sampler Conditions.

Platen Temperature 80 °C Valve & Loop Temperature 150 °C Carrier Pressure 12.5 psi (High Purity Helium) Equilibration Time 30 min Vial Pressurization 8 psi (High Purity Helium) Transfer Line Temperature 200 °C Vial Needle Flow 32 ml/min, helium Pressurize 1.00 min Pressure Equilibration 0.25 min 0.20 min Loop Equilibration 0.20 min 1.00 min Inject

### B. Gas Chromatograph Conditions.

GC Cycle Time

Gas Chromatograph: Varian 3400

Column: JEW DB624 30 m x 0.25 mm x 0.5  $\mu$ m

35 min

Injector Temperature: 175 °C Transfer Line Temperature: (GC-MS) 200 °C

GC Temperature Program Conditions

GC Method:

Initial Value:

Initial Time:

Program Rate:

Final Value:

Time:

DB624

35 °C

2.0 min

8 °C/min

225 °C

7 min

Carrier Gas: High Purity Helium

Source: 80 psi
Column: 12.5 psi
Split Flow: 30 ml/min
Septum Purge: 4 ml/min
Dead time (air 80°C): 83 sec

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(See Figure 2 for GC Operating Parameters.)

# C. Mass Spectrometer Conditions.

Mass Spectrometer: Varian Saturn II Ion Trap Detector

#### Instrument Set Points

Multiplier Voltage: 1400 volts Manifold Temperature: 200 °C Emission Current: 20  $\mu$ amps  $\lambda/M$  Amplitude Voltage: 2.7 volts Integrator Zero: 0.22 average RF Modulator Response: 415 average

### EI/AGC Parameters

EI Background Mass: 50 amu EI Maximum Ioniz. Time: 25000  $\mu$ sec AGC Prescan Ioniz. Time: 100  $\mu$ sec AGC Prescan Storage Level: 125 dacs Data Steps in AGC Prescan: 50 RF Dump Value: 4095 dacs AGC Weight Factor:

### Segment Tune Factors

Segment 1: 110 dacs
Segment 2: 70 dacs
Segment 3: 100 dacs
Segment 4: 90 dacs

# Acquisition Parameters

Acquisition Method: DB624 Mass Range: 40 to 250 amu Seconds/Scan: 0.500 Acquire Time: 18 min Filament/Multiplier Delay: 60 sec Peak Threshold: 2 counts Mass Defect: -50 mmu/100 amu Background Mass: 45 amu Ionization Mode: EI Auto Ion Control: On Cal Gas: Off

#### **APPENDIX G**

STATIC AND DYNAMIC GROUNDWATER CHEMICAL ANALYSIS DATA

Pre-Treatment Static Groundwater Sampling Results (in ppb)

	Depth		Analytes		,	T	1	T	T -			_	-
Location	(ft)	•	Vinyl Chloride	1	1,1,1-Trichloroethan	6	Trichloroethene	;	Toluene	<del> </del>	m+p Xylene	├	
2711	14	BLACK	5.6	<b> </b>	27	1	3.7	1	1 Cilcolio	ND	IIITP AVIOLIA	ND	o-Xyler
2711	16	BLUE	87.5 .		6.5	_	2.2	+	5.2	1.00	<del></del>	ND	<del> </del>
2711	18	RED	1.6		63.7	+	1.1	<del>                                     </del>	43.6		<del> </del>	ND	
2711	22	YELLOW		ND	169	1	1.8	+	148		0.9	שא	l
2721	16	BLUE	3.9	1	19.4	+-	1.0						54.9
2721	18	RED	104	1	109	╅	1	1	50.5			ND	
2731	16	BLUE		ND	2	╅	<del> </del>	1	30.5			ND	
2731	20	WHITE	1.1	1	261	+	28	-	129			ND	<del>.</del>
2731	22	YELLOW	· · · · · · · · · · · · · · · · · · ·	ND	24.2	+	1 1	-	129			ND	
2712	14	BLACK	5.5		4.2	+	1.3	├	<del> </del>			ND	
2712	20	WHITE	27		288	╅	1.3	<del>!</del>	102			ND	
2722	20	WHITE	3.4		84.3	+	1.1		14.9			ND	
2722	22	YELLOW	3		19.4	+-	<del>  '''</del>	<del> </del>	14.5			ND	
2732	16	BLUE	2.5		19.9	+-	<del> </del>		4.8			ND	
2732	18	RED	- 18.6		97.8	+	<del> </del>		84.1	-+		ND	
2732	22	YELLOW		ND	85.1	+	1.2		8.4			ND	
2713	20	WHITE	22		111	┪	1.5	<del> </del>	273			ND	
2723	14	BLACK	38	-	19.4	╫	7.3	-			2.5		37.1
2723	16	BLUE	8		101	+	3.3		9.4 3.8			ND	
2723	16	BLUE	8.4	$\neg$	103	+-	3.3		3.8	-1		ND	
2723	20	WHITE	1.6	<del></del>	509	<del>  -</del>	3.1		160			ND	
2733	14	BLACK	3.9		15.6	┼─	63.5		160			ND	
2733	16	BLUE	1.5		25.4	╁──	03.5		1.1			ND	
2733	18	RED	39.4		127	+-	1.1		170			ND	
2733	20	WHITE		ND	680	<del> </del>	2		139			ND	
2714	14	BLACK	30		13.8	$\vdash$	29	-	12.4	$\rightarrow$		ND ND	
2714	16	BLUE	23.6		16.5	_			12.7		<del></del>	ND	
2714	18	RED	65.7		68.9	1	1.5		51.4			ND	
2714	20	WHITE	3.3		526	_	3.3		2.3	-+	<del></del>	ND	
2714	22	YELLOW		ND	122	_			24.3	-			
2714	22	YELLOW		ND	117	<del></del>	<del></del>		24.3			ND	1.4
2724	14	BLACK	13.4		25.8		10.2		23	-		ND	1.4
2724	16	BLUE	21	$\neg \uparrow$	38.7		3.2			ND		ND	
2734	14	BLACK		ND	10.1	$\vdash$	7.2			ND		ND	
2734	16	BLUE	4.6	-	11.5	$\vdash$	7.2		22	שא		ND ND	
2734	20	WHITE		ND	1420	$\vdash$	5.3		214				
2734	22	YELLOW		ND	275		24			+	27	ND	44.0
2744		Injection	1.2				1.6	$\neg$		ND		ND	44.2
2743		Injection								ND		ND	
2741		Injection	1.4				1.1			VD		ND	
2741		AB DUP	1.4	$\neg$			1.1			<b>8</b>		ND	
2751	E	xtraction	96.3	$\neg$	334	-	2,4	-	529	<del>"" </del>	116	AD .	
2752	E	xtraction	43		528		3.1	$\dashv$	445	$\dashv$	37.3		85.7
2753		xtraction	35.5	7	425		3.5	-+	145	$\dashv$	1.9		54.8
C0619B		200 ppb	181	$\neg \uparrow$	183	-	194	+	197	-	195	-	10.7
C0619C		40 ppb	43.4	_	34.7		40	-	41.5	+		-	204
0619D		200 ppb	191	$\dashv$	195		197	-	204		42.1		43
20619E		40 ppb	42.6	_	35.7		39.6	-+	41.2	∤-	200	- [	201
0619F	1	200 ppb	185	$\neg$	192	$\dashv$	195				41.7	_	43.7
0619G		40 ppb	42.5		35.8	-	39.1	$\dashv$	201		198		198
619H	1	200 ppb	183		181		191		42.1 208	_	41.9	-	42.4
					101				24 H		199		208

ND = None Detected ---= Below Calibration Limit (1.0 ppb) ### = Below Calibration Limit (5.0 ppb)

\* = 50 ppb in QC QC = Quality Control Std

Pre-Treatment Static Groundwater Sampling Results (Continued) (in ppb)

	Dept	nj						T				
Location	(ft)	<del> </del>	1,3,5-Trimethylbenzene		Decane		1,2-Dichlorobenzene	<u> </u>	Undecane	<del>                                     </del>	Napthalene	<del> </del>
2711	14	BLACK		ND	1	ND	1.0	ND	Chacano	ND	Maphialerie	Ň
2711	16	BLUE		ND		ND		ND	<del> </del>	ND		$-\frac{N}{N}$
2711	18	RED		ND		ND	108	+:10	<del> </del>	ND		N
2711	22	YELLOW		ND		ND	652	<del> </del>		ND		
2721	16	BLUE		ND		ND	3.7	<del> </del>	<del> </del>	ND		Ŋ
2721	18	RED		ND		ND	3.9	<del> </del>	-	ND		
2731	16	BLUE		ND		ND	6	<del> </del>	<del> </del>	ND		<u>N</u>
2731	20	WHITE		ND		ND	352	—		ND		N
2731	22	YELLOW	•	ND		ND	44.5	<del> </del>	<del></del>			<u>N</u>
2712	14	BLACK		ND		ND	77.0	† <del></del>		ND		N
2712	20	WHITE		ND		ND	178			ND		N
2722	20	WHITE		ND		ND	170			ND		##
2722	22	YELLOW		ND		ND				ND		##
2732	16	BLUE	···	ND		ND	72.7			ND		N
2732	18	RED		ND		ND	46.1			ND		##
2732	22	YELLOW	<u> </u>	ND		ND	245	-		ND		##
2713	20	WHITE		ND		ND	537	<b>  </b>		ND		##
2723	14	BLACK		ND		ND				ND		##
2723	16	BLUE		ND		ND	<del></del>			ND		##
2723	16	BLUE		ND		ND	<del></del>			ND		N
2723	20	WHITE		ND		ND	173			ND		NE
2733	14	BLACK		ND		ND	1/3			ND		##
2733	16	BLUE		ND		ND	<u>-</u>			ND		NE
2733	18	RED		ND		ND	40.0			ND		NC
2733	20	WHITE		ND		M	49.9			ND		##
2714	14	BLACK		ND		ND	63.7			ND		NC
2714	16	BLUE		ND		ND		***		ND		###
2714	18	RED	·	ND		W	~~~	ND		ND		NE
2714	20	WHITE		ND			76.2			ND		##1
2714	22	YELLOW		ND		ND	20.4			ND		###
2714	22	YELLOW		ND		ND	144			ND		###
2724	14	BLACK		ND		ND	139			ND		###
2724	16	BLUE				ND				ND		ND
2734	14	BLACK		ND		ND	1.9			ND		###
2734	16	BLUE		ND		ND				ND		###
2734	20	WHITE		ND		ND	28.2			ND		###
2734	22	YELLOW		ND		ND	110			ND		###
2744		Injection		ND		ND	562			ΔΛ		###
2743		Injection		ND ND	1.3	<del> </del>				D		ND
2741	$\overline{}$	Injection		ND ND		ND				ND		###
2741	$\neg$	LAB DUP		ND -	1.3	ᄴ	1.8			4D		###
2751	-	Extraction	23.8	וטאי			2			ND		ND
2752		Extraction	7.4		3.8 4.2		538	_	15		52.2	
2753		Extraction					383		20.1		19.9	
0619B	-	200 ppb	187		4.1	•	276		27.7	L		###
0619C	$\neg \dashv$	40 ppb	41.8		40.2	<del>: </del> -	211			•	207	
0619D	-+	200 ppb			40.3		46.9		<u> </u>	•   .	52	
0619E	$\dashv$		193		39.7	•	207		35	•	198	
0619E		40 ppb	42.7		48	•	44.8		42	•	46.8	
0619G		200 ppb	193		50.6	•	206		44.8	•	200	
619H		40 ppb	42.3		51.7	•	45.1			•	48.3	
		200 ppb	192		41.1	•	228			•	223	
0 <u>6</u> 19A		ND		ND		VD		ND				ND

ND = None Detected --- = Below Calibration Limit (1:0 ppb) ### = Below Calibration Limit (5.0 ppb)
\* = 50 ppb in QC QC = Quality Control Std

# Pre-Treatment Dynamic Groundwater Sampling Results (in ppb)

	Depth		Analytes						_			_	,	
Location .	(ft)		Vinyl Chloride	1	1,1,1-Trichloroethane	_	Trichloroethene	├	Toluene		m+p Xylene	<b> </b>		۔
·2711	22	YELLOW		ND		_	2.3		341	<del>' </del>		-	o-Xylene	4
2712	16	BLUE	20.3		25.9	_		├		┼	9.4		97.2	۱.
2721	20	WHITE	57.1		470	-	3.6					ND		<b>-</b> _
2721-dup	20	WHITE	51.8		472			<u> </u>	515	<del> </del> -	3.8	<u> </u>	53.3	J.,_
2722	14	BLACK		ND	17.1		3.6	_	484	<del> </del>	3.7		51.7	ľ
2723	20	WHITE	5.2		436		2.8		3.7	_		ND		N
2724	14	BLACK	10.2	Н			5.7.		197			ND	39.4	Ι.
2731	18	RED	4.6	$\vdash$	31.3		8.2		5.2	L		ND		N
2713	22	YELLOW	11.8		144		1.4		51.3	L		ND		Ň
2732	16	BLUE	1.3	$\vdash$	191		22		696		1.7		28.9	-
2733	14	BLACK	1.3		40.5				7.4			ND		N
2751				ND	16.9		14.7					ND		N
2752		Extraction	111	$\dashv$	503		3		132			ND	<del></del>	"
		Extraction	54		518	Т	3.2		246	$\vdash$	3.6	110		-
2753		Extraction	41.5		575		4		177			ND		-
2C06118A		40ppb		NI	40.5		42.9	_	44.5	-	44.8	וחאו	40.5	-
QC06118B		200 ppb		NI	203	-	193	_	202	$\vdash$			43.5	. <b>_</b>
2C06118C		40 ppb	45.8	$\neg$	35.7	-	39.8			$\vdash$	200		198	
C06118D		200 ppb	238	$\neg$	199	+	197		42.6		42.4		43.3	_
C06119A		40 ppb	47.6	_	35.5	-+			206		202		203	_
BL0618A				ND			38.1		40.8 .		40.7	[	41.7	
				ואט		1D		ND	i	ND		ND		Ñ

	Depth	l		$\mathbf{T}$		T		_	T			_
Location	(ft)		1,3,5-Trimethylbenzene	1	Decane		1,2-Dichlorobenzene	╌	Undecane	├	Nonthalass	+
2711	22	YELLOW		ND		ND	807	+-	Ondocare	ND	Napthalene	_
2712	16	BLUE		ND		ND	80.1	<del> </del>	<del>                                     </del>	ND	<del> </del>	
2721	20	WHITE		ND		ND	353	_	<del></del>	ND		_
2721-dup	20	·WHITE		ND		ND	333	├	·			1
2722	14	BLACK		ND		ND				ND		Į
2723	20	WHITE		ND		ND	236			ND		1
2724	14	BLACK		ND		ND	230	<u> </u>		D		1
2731	18	RED		ND		ND	116			ND		1
2713	22	YELLOW		ND		ND	228			ND		1
2732	16	BLUE		ND		ND	220	-		ND		Į
2733	14	BLACK		ND		ND				ND		I
2751		Extraction		ND		ND	33.2			ND		ļ
2752		Extraction		ND		ND				ND		l
2753		Extraction		ND		ND	1.3			ND		L
C06118A		40ppb	45.4	ND	56.6	חאו	1.5			ND		l
C06118B		200 ppb	194			-	40.8		53.9	اٺ	40	ſ
C06118C		40 ppb	41		50.1		197		49.6	• 1	200	Γ
C06118D		200 ppb	196	-	51.9		45.6		51.4	•	47.2	Γ
C06119A				-+	54.6	•	199	I	49.2	•	186	Γ
L0618A	+	40 ppb	40.5		46	•	43.4		42.3	•	47.3	Γ
200.00				ND		ND		ND		ND		1

# Post-Treatment Dynamic Groundwater Sampling Results (in ppb)

Vial ID#	Location	Depth	Analytes	<del> </del>	444 = 114 = 11							T
1897	11	black	Vinyl Chloride		1,1,1-Trichloroethan	_	Trichloroethen	е	Toluene		m+p Xylene	<b>,</b>
1898	11	blue	<del> </del>	ND		ND				ND		NE
1900	11	white	<del> </del>	ND		ND				ND	T	NE
1901	11 -	yellow	<del>                                     </del>	ND		ND		ND		ND		NE
1902	21	black	ļ <u>.</u>	ND	66.1	1	2.8		175	T	2.4	7
1903	21	blue	<del> </del>	ND		ND	9.7			ND		NE
1904	21	red	<u> </u>	ND		ND	2.6			ND		IND
1905	21	white	<del> </del>	ND		ND	2.3	Ĺ		ND		ND
1906	21	yellow		ND		ND	3.6			ND		NE
1907	31	black			25.7	<u> </u>	2.9		33.8	T		
1907 DUP	31	blue		ND		ND	4.2	L		ND		ND
1908	31		<del> </del>	ND		<del> </del>	4.4			ND		ND
1909	31	red white		ND		ļ <u></u>	5.7			T		ND
1910	31			ND				***		ND		ND
1911	12	yellow black		ND		ND						
1912	12			ND	24.4	<u> </u>	11.4		80.7			=
1913	12	blue		ND		<u> </u>	10.3			ND		ND
1914	12	red white		ND		ND		ND		ND		ND
1915	12			ND						ND		ND
1916	22	yellow black		ND		ND	3.2			ND		ND
1917	22			ND	3.9		3.4		59.1		1.9	Ι
917 DUP	22	blue		ND		ND	3,1			ND		ND
1918	22	red		ND		ND	3.1			ND		ND
1919	22	white		ND		ND				ND		ND
1920	32	yellow		ND		ND			-	ND		ND
1921		black		ND			2.3			ND		ND
1922	32	blue		ND	5.5		5.3		7.7			ND
1923	32	red		ND						ND		ND
1924	32	white		ND								ND
1925	32	yellow	·	ND		ND		ND		ND		ND
1926	13	black				ND		ND		ND		ND
1927	13	blue			14.1		7.8		19.3			ND
927 DUP	13 23	white		ND	<u> </u>	ND				ND		ND
1928	23	black		ND		ND				ND		ND
1929	23	blue		ND		ND		ND		ND		ND
1930	23	red		ND		ND	4.6			ND		ND
1931	23	white		ND		ND						ND
1932	33	yellow		ND		ND		ND				ND
1933	33	black		ND		ND		ND		ND		ND
1934	33	blue		ND		ND		ND		ND		ND
1935		red			15.4		4.1		51.5		7.1	
1936	33 33	white		DV						ND		ND
1937		yellow		ND						ND		ND
1938	14	black		-				ND		ND		ND
1939	14	blue		ND		ND		ND		ND		ND
39 DUP	14	white		1D	10.4		6.8		1.5	-		ND
	14	yellow		1D	10.3		6.6		1.5	_		ND
1940 1941	24	black		1D		ND		ND		D		ND
1943	24	red		ID		ND		ND		ND		ND
	24	white		ID				ND		VD		ND
1944	34	black	1.5		153		3.1		10.8	<del></del>		<u>"</u>
1951	34	blue		D		VD		ND		ΙŌΙ		ND
1953	34	red		ID		VD		ND		ND		ND ON
1954	34	yellow				VD		ND		_		ND ND
1956	51		N	D			1.6	-		VD		뛺
1957	52	·				ND				ND		뛺
1958	53			<u>-</u>		VD		D		1D	——————————————————————————————————————	

# Post-Treatment Dynamic Groundwater Sampling Results (Continued) (in ppb)

Vial ID#	Location	Depth	o-Xylene	i i	1,3,5-Trimethyfbenzene	a i	Decane	$\overline{}$	1.2 Diablasshassas		100-4	+	
1897	11	black		-		ND	Docare	ND	1,2-Dichlorobenzen	₽	Undecane	Napthalene	
1898	11	blue		ND		ND		ND	<del> </del>	<del> </del>	NI NI		1!
1900	11	white		ND		ND	<del> </del>	THE DIA	<del> </del>	ND	NI NI		
1901	11	yellow	44.7	-	1.2	110	<del> </del>			2.8	NC NC		] [
1902	21	black		ND		ND	<del> </del>	ND	3.7	-	l NE		1
1903	21	blue		ND		ND.	<del></del>	ND		ND	NO	<u>л</u> — —	Ţ~~ <b>ī</b>
1904	21	red	1	ND				ND		ND	NC		1
1905	21	white	<del> </del>	140		ND		ND		ND	NE	<u>,                                     </u>	1
1906	21	yellow	58.3	-		ND		ND	4.8	1.	NC	,	<del> </del>
1907	31	black	36.3	ND	·	ND		ND	834	Τ-	ND		
907 DUP	31	blue				ND		ND	1.5	1 -	ND		- 1
1908	31		ļ	ND		NO		ND			ND		-
1909		red		NO		ND		ND		1	ND		
	31	white		ND		ND		ND					
1910	31	yellow	1.1			ND		ND	19.7	<del> </del> -	ND ND		11
1911	12	black	70.1			NO		ND		-	ND		1
1912	12	blue		ND		ND		ND	768	<del>                                     </del>	ND ND		
1913	12	red		ND		ND			1,1	1—1	ND		_ N
1914	12	white		NO	······································			ND			ND		N
1915	12	yellow		ND		ND		ND			ND		Ī
1916	22	black	15.6	NU	<del></del>	9	1.4		1.3			<del> </del> -	
1917	22	blue	15.6	<del></del> 1				ND	4.4		ND	<del> </del> -	
17 DUP	22			ND		ND		ND		ND	ND ND	<del>                                     </del>	
1918	22	red		ND		ND		ND		ND	ND		<del>-</del> 7
1919		white		ND		ND		ND			ND ND	<del> </del>	
	22	yellow		ND		ND		ND	·	ND		<del>  </del>	N
1920	32	black		ND		ND		ND			ND	<b></b>	N
1921	32	blue	27.7			ND		ND	2.3	-	ND.	I	N
1922	32	red ·		ND		ND		ND	2.0		ND		<u>N</u>
1923	32	white		ND		ND		ND		NO	ND		_ N
1924	32	yellow		ND	~ <del>~~~</del>	ND			~	ND	ND		N
1925	13	black		ND		ND		ND		ND	ND ND		N
1926	13	blue	17.6	<del>.,,,</del>				ND			ND		N
1927	13	white		NO		ND		ND	324	1	ND		-
7 DUP	23	black		ND		ND		ND			ND		Ň
1928	23	blue		ND		ND		ND			ND		NI
929	23	red				ND		ND			ND		N
1930	23			ND		ND		ND			ND		N
931	23	white		ND		ND		ND		ND	ND		
932		yellow		ND		ND		ND			ND	<del></del>	<u>N</u> !
933	33	black		ND		ND		ND			ND		<u>N</u> I
	33	blue		ND		ND		ND	1				N
934	33	red	96.4	$\Box$	17.2			ND	718		ND	<del></del>	N
935	33	white	1	VD		ND -		ND	/ 10		ND	41.1	
936 .	33	yellow	1	OV		ND		ND			ND		NC
937	14	black		vo l		ND					ND		NC
938	14	blue		<del>100</del>		ND ON		ND		ND	ND		NE
939	14	white		VD -				ND	1.1	i	ND		NC
DUP	14	yellow		10		ND		VD.	3.1	$_{-}$ T	ND		NE
940	24	black				ND		AQ.	2.9		ND	<del></del> +-	ND
941	24	red		ID		ND		9		ND	ND		- ND
43	24			D		ND		VD		ND	ND	<del></del> -	ND
44		white		ID				4D	1.2	-	ND ND	<del></del>	
	34	black	30.9	$\perp$		ND		ND	3	~	- ND	1	ND
951	34	blue	N	D		NO		io		ND -		- 1	ND
953	34	red	N	D		vo l		1			ND ND		_ND
354	34	yellow	Ň	D		QV	3.8	<del>" -</del>		ND	ND		ND
56	51		N			<del>6</del>		<del></del>		ND	ND		ND
57	52		N			<del>6  </del>		D :	1.3				ND
58										OV	ND		

# Post-Treatment Static Groundwater Sampling Results

			Analytes	T	T	<b>T</b>			<del></del>	<del>-,</del>				
Vial ID#	Location	Depth	Vinyl Chloride	_	1,1,1-Trichloroethane	-	Trichloroether	_	Toluen	-	- V.d	<del>-</del>	<del> </del>	
3762	2711	black		ND	11117	NI	14.7	- 18	1 Oluen	ND	m+p Xylen		o-Xylene	
3763	2711	blue		ND	r	N			+	ND		ND ND	<del> </del> -	ND
3764	2711	red	~	ND		N		╅	<del></del>	1,40	<del> </del>	ND		ND
3765	2711	white		ND		NE		1	1.1	$\dagger$	<del></del>	ND	<del></del> -	
3766 3767	2711	yellow	<del> </del>	ND	24.1		5.8	$\top$	150	1-	1.4	+	67.5	<b>∤</b> =-
3768	2721 2721	black	<del> </del>	ND		NE				ND	<del>                                     </del>	ND	<del></del>	ND
3769	2721	blue	<del> </del>	ND		NE				ND		ND		ND
3770	2721	red	· · · · · · · · · · · · · · · · · · ·	ND		NC				ND		ND		ND
3771	2721	white	<u> </u>	ND		NE					1.8	1	1.1	
3771 DUP	2731	yellow	<del> </del>	ND	11.6	_	5.2		239		86.6		129	- · ، ا
3772	2731	black	<del> </del>	ND	11.7	Щ.	5.3		239		64.4		131	†
3773	2731	blue	<u> </u>	ND		ND				ND		ND		ND
3774	2731	red white	<del> </del>	ND	-	ND	8.6			ND		ND		ND
3776	2731	vellow	ļ	ND		ND						ND		ND
3777	2712	black		ND	8.5	<u> </u>	15.2		35		3.1		59.2	1 :==-
3778	2712	blue	<u> </u>	ND		ND	12.1			ND		ND		
3779	2712		<del> </del>	ND	<del></del>	ND	7.7			ND		ND		ND
3780	2712	white		ND		ND	4.8	1_		ND		ND		ND
3781	2712	vellow	<del>  </del>	ND	- 10	ND	6.9	<u> </u>		ND				
3782	2722	black		ND ND	1.8		9.6	-	36.2		3.4		37.9	
3782 DUP	2722	blue		ND		ND	15.1	<b>└</b>		ND		ND		ND
3783	2722	red		IND	· · · · · · · · · · · · · · · · · · ·	ND	14.2	<del> </del>		ND		ND		ND
3784	2722	white		ND		ND		ND	6.8	ND		ND		ND
3785	2722	vellow		ND		ND	<u> </u>			ND		ND		ŅD
3786	2732	black		ND	1	עא	8.5	<del>ا</del>		ND		ND		ND
3787	2732	blue		ND		ND	13.8 7.8		7.8				24.5	
3788	2732	red		ND		110	8.1	-		ND		ND		ND
3789	2732	white		ND		ND	0.1	-		ND		ND		ND
3790	2732	yellow		ND		ND	1.9					ND		ΝĎ
3791	(red)	black		ND	4.1		14.4		38.5		5.3			
3792	(white)	blue		ND		ND	2.6		30.3	ND	5.3	ND	32.6	
3792 DUP	(yellow)	white		ND		ND	2.8			ND		ND -		ND
3793	2723	_black		ND		ND	8.2			ND		ND		ND ND
3794 3795	2723	blue		ND		Z	13.6					ND		닒
3796	2723	red		ND		ND	10.4			ND	<del></del>			
3797	2723 2723	white		ND		ND	6.4					ND		ND
3798	2733	yellow		(D)		ND	1.5			ND		ND		ND
3799	2733	black		4D		ND	3.3			ND		ND		ND
3800	2733	blue		ND	2.7		9.2		46.7		10.5		64.2	-392
3801	2733	red white		ᇷ		ND	11.1			ND		ND		ND
3802	2733	yellow		1D		ND	6					ND		ND
3802 DUP	2714	black		1D		ND	-			ND		ND		ND
3803	2714	blue		ID		ND				ND		ND		ND
3804	2714	red		ID	2.4		2.6							
3805	2714	white		<u>10</u>		-	14.2		27.8		10.3		36.1	
3806	2714	yellow		ы		ND	2.4			ND		ND		ND
3807	2724	black		ib		ND ND				ND		ND		ND
3808	2724	blue		Ď		וסא		ND		=-	:		1.2	
3809	2724	red		D -	44.1	40	5.6	ND		ND		ND		ND
3810	2724	white		<u></u>		VD	4.1	$\dashv$	286	<del></del>  -	12.2		140	
3811	2724	yellow		D		<del>"</del>	4	$\dashv$		ND		ND		ND
3812	2734	black	1.3		1.4	_		_	1.3	=+		=		
812 DUP	2734	blue	1.2	J	1.3	1		_+	1.2			ND		ND
3813	2734	red			4.7	$\dashv$	4.7	$\dashv$	1.3			ND D		ND
3814	2734	white		D	6.2	_	5.2	+	4.5	-+		ND	33.7	
3815			N		3.2		9.9	_		1D		10		ND
3816			N			1D	1.4	$\dashv$		<u>101</u>		D		#B
3817 3818			N			ID		ND		io		ib		器
3819	2734		N			iD	1.5			-		io -	1 ' '	۳'
3820	2751	yellow	N	ן ט	72.8		7.7		265	$\neg$	86.7	-	109	
3821	2752		25.7		66.6		2.2	$\Box$	78.1		3.4	$\top$	37.7	1
3822 .	2753		25.2		52.6		2.3	$\Box$	43.7	J	1.7		27.7	
	4/00		2.6	_L	72.7	- 1	2.8	T	50.7	-7-	-		10.8	- 1

# Post-Treatment Static Groundwater Sampling Results (Continued)

		T		<del>T :</del>	T .	T-	T		1			
Vial ID#	Location	Depth	1,3,5-Trimethylbenzene		Decane	+	1,2-Dichlorobenzene		Undecane	+-	Napthalen	<del>.  </del>
3762	2711	black		ND		ND		ND	- Chaocain	ND	Hapmalen	ND
3763	2711	· blue		ND		ND	T		1	ND	<del> </del>	ND
3764	2711	red		ND				ND	<del> </del>	ND		###
3765	2711	white				ND				ND	T	###
3766 3767	2711	yellow	19.5	1		ND	: 559				71.8	7
3768	2721 2721	black	<u> </u>	ND		ND				ND		###
3769	2721	blue		ND	···	ND				ND	I	ND
3770	2721	red		ND		ND	·	<u> </u>		ND		ND
3771	2721	white	1.3	<u> </u>	4.5	ļ	15.6	L		ND	5.4	
3771 DUP	2731	yellow	34.7	ļ.,	<u> </u>	ND	1010			ND	112	]
3772	2731	black	24.1	<del> </del>		ND	998	<u> </u>		ND	116	7
3773	2731	blue		ND		ND				ND		###
3774	2731	red		ND		ND	1.1			ND		###
3776	2731	white		ND		ND				ND		ND
3777	2712	yellow	22	l		ND	519			ND	18.4	ND
3778	2712	black blue		ND		ND	1.1			ND		ND
3779	2712			ND		ND	<u> </u>			ND		ND
3780	2712	red white		ND		ND				ND		###
3781	2712	yellow	15.7			**		ND		ND		###
3782	2722	black	10./	ND			361			~~	421	1. 🗀
3782 DUP	2722	blue		ND		<b>2 2</b>				ND		###
3783	2722	red		ND		22 25				ND		ND
3784	2722	white		ND		W W				ND		ND
3785	2722	yellow		ND		ND		ND		ND	·	###
3786	2732	black	· · · · · · · · · · · · · · · · · · ·	140		ND	166			ND		ND
3787	2732	blue		ND		ND	100			ND		###
3788	2732	red		ND		ND		=		ND		ND
3789	2732	white		ND		ND		_		ND		###
3790	2732	yellow				ND	3.2			ND		ND
3791	(red)	black	8.8			ND	376	-		ND ND	<del></del>	###
3792	(white)	blue		ND		ND				ND	7.8	
3792 DUP	(yellow)	white		ND		ND				ND		ND ND
3793	2723	black		ND		ND				ND		ND
3794	2723	blue		ND		M		ND	5.7			###
3795	2723	red		ND		ND	1.8			ND		ND
3796	2723	white		ND		ND				ND		ND
3797	2723	yellow		ND		M		ND		ND		ND
3798	2733	black			3.2			ND		ND		###
3799	2733	blue	16.4			ND	718		2.9		55	"""
3800	2733	red		ND		ND	<u> </u>			ND		ND
3801	2733	white		ND		ND				ND		ND
3802	2733	yellow		ND		ND		ND		ND		ND
3802 DUP	2714	black		ND		ND				m		ND
3803	2714	blue				<b>^</b>		ND		ND		###
3804	2714	red	13.5			ND	353			ND	13.2	
3805	2714	white		ND		ND		ND		ND		ND
3806 3807	2714	yellow		ND		ND		ND		ND		ND
3808	2724 2724	black				<del></del>		ND		ND		ND
3809	2724	blue		ND		M		ND		ND		ND
3810	2724	red white	23.3	ND -		M	\870			<b>M</b>	62.4	
3811	2724	yellow		ND		ND		ND		ND		ND
3812	2734	black		ND ND		ND		ND		ND	]	ND
812 DUP	2734	blue		ND ND				ND .		ND		ND
3813	2734	red		ND	<u>-</u>  .	-		ND		ND		ND
3814	2734	white	2.1	ואוי			4.1	-		ND		ND
3815		44,117		ND		ND DV	445			VD		ND
3816		<del></del>		ND ND		AD -	1.5			ND		ND
3817	<del></del> -	<del></del>		ND		10		ND		ND		ND
3818		<del> -</del>		ND D	2	ND		D		ND		ND
3819	2734	yellow	16.7	140		8	29.7	-		VD .		ND
3820	2751	75511	9				744			ND	30.6	
3821	2752		6.4	-+-		4D	315	+	2.6		19.6	
3822	2753			ND	F	AD	224			1D		###
				ושאו		עט	132			1D		ND

# APPENDIX H

LOW-FLOW GROUNDWATER SAMPLING LOGS

		•	10-2741 nel <u>T. p/c.4/</u> M. G/L	ME 5.6	e Water 🖸 سروع مروع, Date		d Water ⊠			ication $U1-2741$
-	Time_ <i>[0</i>	<u>49       </u> ing Deptl	SUMMARY: Depth to Wa h_27.78 TOC	_ Bore	De Diamet	er	uctCalc	ulated Pur	ge Volume	5.0 Gallons
	SAMPLING Sampli Pump	ng Metho	ARY: Producte  Od: Dedicate  Vi:00 Pur	ו אייייי כ d Bladder np Stoppe	Pumo	Portable	Bladder Purr	np	Bailer	lead
	Time (military)	pН	SC (umhos/cm)	Temp	Turbiditý (NTU)	Salinity	Vol ( <del>gal.)</del> Evac.		DO	Comments
	11:01	6.96	381	22.4						
	11:02	6.96	369	27.3			2	<del></del>		
	11:03	6.97	364	22 1			3			
	11:04	6.97		22.0			4			
	11:0.5	6,96	353					•		COLLECT
•	11:23	6.94	290	21.8			14		•	FINAL
	<u> </u>									
			<del></del> .							
				<del></del> .						
				<del></del> .	· · · · · · · · · · · · · · · · · · ·					
			<del>-,</del> -			<del></del>		•		
	<del></del> .	· ·		<del></del> .	··	<del></del> .	<del></del>			
									•	
8/1/95	INSTRUMEN	ITATION	l: Orion Fl	SAVER.	X Reference	Solution				
_	•	• •				ffers: . 4 [		10 🗆		
2208.121					lution		] 7[] ·	10 LJ		
22						_umnos/cm NTU:				1
ğ			· · · · · · · · · · · · · · · · · · · ·		e conniou -		, ;			
PROJECT NO.	TIME (1:10	VOCs_		BNAEs_	X : Bs_0N-4.	PEST//c/ Metals/Cation	ons	ТРН	火	_

HILL AIR FORCE BASE
OPERABLE UNIT 1
TREATABILITY STUDY
GROUND-WATER SAMPLING LOG
FIGURE A-18

	Location	nel	Surface	Water Date		d Water □ /96		•	ication <u>U1-275</u>
Time/_ Total Ca	2:360	SUMMARY: Depth to Wa h_23, 49 TOC	Bore	21De hole Diamete inal pH_6_	- 10		ulated Pu		So Gallons (°C) 2 4 5
SAMPLING Samp Pump	ling Metho	ARY: PE od: Dedicated /250 Pun	d Bladder	TTC PUMP	Portable	Bladder Pum s <u>/ン</u> Org	p	Bailer	ead
Time (military)	pH 	SC <del>(umhos/cm)</del>	Temp (°C)	Turbidity (NTU)	Salinity	Vol <del>(gal.)</del> Evac.	Eh	DO	Comments
1251	6.95	263	24.3			1	<del></del>	-	
1252	6.95	262	24.3			2			• • • • • • • • • • • • • • • • • • • •
1253	6,95	271	24.4			3		•	
1254	6.95	278	24.4			<del></del>		• ———	
1255	6.94	278	24.4	<del></del>	· .		-	·	Court
1305	6.94	265	24.5			12.			SANGLES FIRM
1 EM.	VEKC -	JATER PU	ETER.	VIS BUC	EET MEA	SURED AT	16.5	- VSI	V6
INSTRUME		Horiba SC Refe Turbidity	☐ pH Ca	lution se Solution _	fers: 4 [ _urnhos/cm NTUs				
TIME 12:5.	_vocs	<u> </u>	BNAEs _	<u> </u>	BsT/PC/ Metals/Calid	ons X	TPH	1 <u>X</u>	<b>-</b>

HILL AIR FORCE BASE
OPERABLE UNIT 1
TREATABILITY STUDY
GROUND-WATER SAMPLING LOG
FIGURE A-18

Time / 2	ing Depth	SUMMARY: Depth to Wat 23.65	Borel	nole Diame	ter	Calc	ulated Pur	duct Thickn ge Volume Final Temp(	ess
SAMPLING Sampli	ng Metho	ARY: Pe d: Dedicated 1239 Pur	l Bladder	Pump	Portable	Bladder Pum	p	Bailer	
Time (military)	pН	SC (umhos/cm)	Temp (°C)	Turbidity (NTU)	Salinity	1175	janic Vapo Eh	DO DO	ead Comments
1240	6.94	285	23.8			1			
1241	6.93	282	24.7		·	2.		<del></del>	<del></del>
1242	6.93	274	24.8	<del>~</del>		3,			
1243	6.94	279	24.6		<del></del>	4	•		
. 12 44	6.94	275	24.5	• • • • • • • • • • • • • • • • • • • •		5			
1245	6.94	279	24.6			6			COLLECT EPH SELDS ONLY
		<del></del>							
		<del></del>	<del></del> -					• .	
			• 1						
							·····	• •	
								· ·	
INSTRUMEN	NOITATION	Horiba SC Refe	pH Ca	lution	Solution uffers: - 4   _umhos/cm	l .	0 🗆		

HILL AIR FORCE BASE
OPERABLE UNIT 1
TREATABILITY STUDY
GROUND-WATER SAMPLING LOG
FIGURE A-18

	Sample Samplin		<i>U</i>  -27 <i>5</i> ]	Surface	Water □ Date	Ground   6   21	d Water 🔯			cation <u>VI - Z7.5</u>			
	Time 12 Total Cas	14_ ing Dept	SUMMARY: Depth to Wa h_23.89 TOL	Borel	S Dep	r	Calc	ulated Pur	duct Thickn ge Volume Final Temp(	5.3 Gallons			
	SAMPLING Sampli Pump	ng Metho	ARY: od: Dedicated   217_ Pun	d Bladder	Pump d_ <i>[235</i> T	_ Portable l ບານຂອງ otal <del>Gallon</del> s		p	Bailer				
	Time (military)	pН	SC (umhos/cm)	Tēmp (°C)	Turbidity (NTU)	Salinity	Vol (gal.) Evac.	Eh	DO	Comments			
	1218 1219 1220 1221 1222 1223 1235	6.94	282 281 273	23.3 24.1 24.1 24.1 24.3 24.1 24.5			1 2 3 4 5 6 15			COLLOCT SAUDIFY FINAL			
	INSTRUMENTATION: Orion Eh Meter Reference Solution  Horiba												
Π!	ME <u>1663</u>		Itur X	BNAEs _	X	Metals/Cation	ons XX	TPH	<u>×</u>				

HILL AIR FORCE BASE
OPERABLE UNIT 1
TREATABILITY STUDY
GROUND-WATER SAMPLING LOG
FIGURE A-18

PROJECT NO. 2208.1211 8/4/95

	Sample Loca	ation <u>vi- 2744</u>	Surface	Water []	Groun	d Water 🔯			cation <u>01-2744</u>
Ļ	- Camping C			. Date	. 10/21/	7.6	Weathe	r Junny	1.T≈40°F
	Time_1/48	Depth 23.95	Borel	De Diamete	er		ulated Pur	duct Thicknorge Volume	5.7 Gallons
	SAMPLING SUI	MMARY:					· · · · · · · · · · · · · · · · · · ·		
	Sampling Method: Dedicated Bladder Pump Portable Bladder Pump Bailer Pump Started Pump Stopped Total Gallens Organic Vapor at Well Head								
	1	oH SC (umhos/cm)	Temp (°C)	Turbidity (NTU)	Salinity	Vol (gal.) Evac.	Eh	DO	Comments
		194 294	17.4			$\overline{\Box}$			
		94 293	20.1		· ·.	_2			
		294.	20.6				<del></del> :		
		93 295	20.7	<del> </del>		_4	<del></del>		
	1	.94 288	21.0		·	_5	•		
		94 288	21.0	· · · · · ·	<del></del> :	<u>6.</u>			(occet (AMAE)
	1211 6.	93 269	21.5		<del></del> -	15			FINAL
	<u> </u>		<del></del> -	<del></del> .		·	<del></del>		
					·				
					<del></del>	<del></del> -	` `		
					· 				
		<del></del> -	·		<del></del>			·	
				· .	<del></del> .		· ·	·.	
			<u>:                                      </u>	<del>· · ·</del> -					
	L		:		<del></del>			·	
11 8/4/95	INSTRUMENTATION: Orion Eh Meter Reference Solution  Horiba								
0. 2208 1211	SC Reference Solutionumhos/cm Turbidity Reference SolutionNTUs								
PROJECT NO.	TIME 12:00 VO	OCs X Diop/E/R.		X1	Post/fcg Metals/Catio	ens X	ТРН	X	-

HILL AIR FORCE BASE OPERABLE UNIT 1 TREATABILITY STUDY GROUND-WATER SAMPLING LOG FIGURE A-18

ų.

PROJECT NO. 2208.1211 8/4/95

Sample	Location	VI-2743	Curloss	Water F7					
Sample Location v1-2743 Surface Water Ground Water Sample Identification v1-2743  Sample Personnel T. M. Uhr. F. J. G. IV. Date 10/21/16 Weather SUNY TE 40°F									
Sampling	y reison	M. GILDE	7 3.6W 4	v, Date	10/21/3	6	Weathe	<u>r Sunny</u>	, T≈40°F
MEASUREMENT SUMMARY:  Time 1053 Depth to Water 15.26 Depth to Product Product Thickness  Total Casing Depth 23.42 Borehole Diameter Calculated Purge Volume 4.9 Gallions  Measuring Point 100 Final pH Final SC Final Temp(°C)									
SAMPLING SUMMARY:  PERSTACTIC PUMP  Sampling Method: Dedicated Bladder Pump Bailer Bailer Pump Started I1:29 Pump Stopped									
Time (military)	рН	SC (umhos/cm)	Temp	Turbidity (NTU)	Salinity	Vol (gal.) Evac.	Eh	DO	Comments
11:30	6.95	295	21.1						
11:31	6.94	293	21.3		·	2		~	-
11:32	6.93	288	21.5			3			
11:33	6.93	284	21.5	-		4			
. 11:34	6,93	283	21.4			5			COLLETT SAMPLES
11:46	6.93	269	21.3			12			FINAL
					•				
				•					
		•							
				-					
								······································	
		<del></del>				<del></del>	······································		
				<del></del> -	<del></del>		· · · · · · · · · · · · · · · · · · ·		·
		Sugar.	SAVER	×					
NSTRUMEN	MOITATE	V: Orion El		Reference	Solution				
Horiba ☐ pH Calibration Buffers: 4 ☐ 7 ☐ 10 ☐									
SC Reference Solutionumhos/cm									
Turbidity Reference SolutionNTUs									
						<u> </u>			
IME 11:35	VOCs	_X	RNAFe	χ -	PET / PC	3	TDU	X	
		<del></del>	DI 14472		iviolal <del>s/Call</del>	- A	IFM	<del></del>	<del></del>

HILL AIR FORCE BASE OPERABLE UNIT 1 TREATABILITY STUDY GROUND-WATER SAMPLING LOG FIGURE A-18

#### APPENDIX I

#### LIMITATIONS FOR STEAM INJECTION TO MOBILIZE RESIDUAL NAPL CONTAMINATION

# LIMITATIONS FOR STEAM INJECTION TO MOBILIZE RESIDUAL NAPL CONTAMINATION

Steam injection has been applied successfully in enhanced oil recovery (EOR) for over 40 years (Chu, 1985). Early applications of steam injection emphasized the heating of heavy oils by sweeping steam across the top of the reservoir. In this scenario, heat from the steam is conducted downward into the oil bearing zones. The heat reduces the viscosity of the oil and increases the percentage of the oil originally in place which drains downward by gravity into recovery wells. The fraction of the original oil left in the reservoir often remains relatively high (>50%) but further recovery is not economical. Obviously, this approach is unacceptable in environmental applications where greater than 90% recovery is sought. More recent applications of steam injection for EOR have targeted lighter oils (Blevins et al., 1984) which may be amenable to a steam drive. Yet, laboratory studies, computer simulations, and field projects indicate the primary recovery mechanism for light oils is distillation rather than a pressure-gradient push of the oil. A model for the steam drive of a residual oil was developed by Stewart and Udell (1988) and compared with laboratory and field data further substantiating this conclusion. In the development which follows, the model of Stewart and Udell is used to determine an expression for the limiting viscosity of a NAPL allowing its displacement in a steam drive. The limiting viscosity is then determined for the conditions found at Operable Unit 1, Hill Air Force Base, Utah.

#### 1. MODEL DEVELOPMENT

Consider a one-dimensional, linear porous medium saturated with water and a residual non-aqueous phase liquid (NAPL). The initial NAPL in place is assumed to be uniformly distributed in the matrix and residual is defined to mean the NAPL is immobile in a waterflood. The medium is initially at temperature  $T_{amb}$  and is subjected to steam injection at a constant mass flux and enthalpy at x = 0. For a constant steam injection rate and quality, the problem becomes quasi-steady, i.e., the steam condensation front progresses at a constant velocity  $(V_f)$ . If capillary effects are neglected and the heated zone ahead of the steam condensation front is small, three regions form. First is the steam zone where the NAPL is assumed to be completely displaced. Second is the condensate and NAPL bank being pushed by the steam flow. Third is

the residual NAPL zone which has yet to be impacted by the steam. The NAPL bank grows at a faster rate than the steam condensation front since it is assumed to be completely displaced. In formulating the problem, the following assumptions are made for each of the three regions:

- 1. Fluid densities are constant:
- 2. The porous medium is incompressible and homogeneous;
- 3. All flow is dominated by viscous forces, allowing the use of Darcy's law;
- 4. The temperature gradient in the steam zone is small so that heat conduction can be neglected;
- 5. Capillary pressure, relative permeabilities, and effective thermal conductivity are single-valued functions of the local wetting phase saturation;
- 6. Heat of vaporization, interfacial tensions, and fluid viscosities are constant;
- 7. The water phase is wetting while the steam vapor and NAPL are non-wetting; and
- 8. The NAPL and water are effectively immiscible.

The multiple zones are illustrated in Figure I-1. The NAPL is considered non-volatile and complete steam displacement of the NAPL is assumed to determine the limiting conditions for the contribution of viscous forces to NAPL recovery during a steamdrive. Because of different heat- and mass-transfer mechanisms, the regions are formulated separately and coupled through appropriate boundary conditions at each interface. A more detailed development of the model presented below can be found in Stewart and Udell (1988).

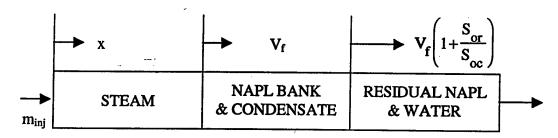


Figure I-1. Idealized Steam Drive.

### 1. Requirement for Stable NAPL Displacement by Steam Flow

A marginal stability criterion was derived by Saffman and Taylor (1958) for the immiscible displacement of oil by water in a porous medium. The criterion required that the pressure gradient in the injected water equal or exceed the pressure gradient in the mobile, displaced oil. If this condition is not met, then the water fingers through the oil and much of the oil is left behind. Similar criteria have been developed for miscible displacements and steam

displacing water. In the current scenario, steam vapor and the NAPL are considered non-wetting and the marginal stability criterion for stable displacement of the NAPL becomes:

$$-\frac{dP_{v}}{dx} \ge -\frac{dP_{o}}{dx} \tag{I-1}$$

where P represents the pressure and the subscripts v and o designate the steam vapor and NAPL phases, respectively. Assuming horizontal displacement and substituting Darcy's law for the pressure gradient in (I-1) yields:

$$\frac{m_{\nu}\mu_{\nu}}{\rho_{\nu}k_{r\nu}k} \ge \frac{m_{o}\mu_{o}}{\rho_{o}k_{ro}k} \tag{I-2}$$

where m is the mass flux,  $\mu$  is the viscosity,  $\rho$  is the density,  $k_r$  is the relative permeability, and k the absolute permeability. Behind the steam condensation front, the vapor mass flux is given by:

$$m_{v} = m_{ini} X \tag{I-3}$$

where m<sub>inj</sub> is the mass flux of the injected steam and X is the steam injection quality. Conservation of mass applied to the NAPL phase yields the mass flux of the NAPL:

$$m_o = \rho_o \phi V_f \left( 1 + \frac{S_{or}}{S_{oc}} \right) \left( S_{oc} - S_{or} \right)$$
 (I-4)

where  $m_0$  is the mass flux of the displaced NAPL,  $\rho_0$  is the density of the NAPL,  $\phi$  is the total porosity of the soil,  $V_f$  is the velocity of the steam condensation front,  $S_{or}$  is the residual NAPL saturation, and  $S_{oc}$  is the constant NAPL saturation in the NAPL bank being pushed by the steam. The velocity of the NAPL bank front is  $V_f$  (1+ $S_{or}/S_{oc}$ ). Because capillary effects are neglected, the saturations are constant in each of the three regions and discontinuous at the

interfaces. Capillarity serves to smoothen the discontinuities and usually occurs over relatively short intervals. The saturations are illustrated in Figure I-2 where the wetting and non-wetting phase saturations must add to one in each region.

1	STEAM	NAPL BANK & CONDENSATE	RESIDUAL NAPL & WATER
1		·	$S_o = S_{or}$
		$S_o = S_{oc}$	
	$S_{v} = 1 - S_{c}$		0 -1 0
	·	$S_{w} = 1 - S_{oc}$	$S_{w} = 1 - S_{or}$
0	$S_1 = S_c$		

Figure I-2. Saturation Distributions

Substituting (I-3) and (I-4) into (I-2) yields:

$$\frac{m_{inj}X}{\rho_w\phi V_f} \ge \frac{\mu_o \rho_v k_{rv}}{\mu_v \rho_w k_{ro}} \left( 1 + \frac{S_{or}}{S_{oc}} \right) \left( S_{oc} - S_{or} \right) \tag{I-5}$$

As described above, the relative permeabilities are solely functions of the fluid saturations. Therefore, it remains only to develop expressions for the liquid saturation in the steam zone  $(S_c)$ , the NAPL saturation in the displaced bank  $(S_{oc})$ , and the condensation front velocity  $(V_f)$ .

#### 2. Liquid Saturation in the Steam Zone

For horizontal, one-dimensional flow, the vapor saturation in the steam zone is solely a function of the steam injection quality. If the quality is one, the vapor saturation is equal to one minus the irreducible water saturation (i.e., capillary condensation maintains a limited liquid saturation where soil grains contact each other). From multiphase flow theory, equating the pressure gradients in the steam liquid and vapor phases and using Darcy's law yields an expression for the vapor saturation in the steam zone:

$$-\frac{dP_{\nu}}{dx} = -\frac{dP_{l}}{dx} \tag{I-6}$$

$$\frac{m_{inj}X\mu_{v}}{\rho_{v}k_{rv}k} = \frac{m_{inj}(1-X)\mu_{l}}{\rho_{l}k_{rl}k}$$
 (I-7)

where I represents the liquid phase in the steam. Rearranging yields:

$$\frac{k_{rl}}{k_{rv}} = \frac{(1-X)\mu_l \rho_v}{X\mu_v \rho_l} = \Psi$$
 (I-8)

The left-hand-side of this expression is solely a function of the saturation while the right-hand-side is a constant. For this investigation, the relative permeability functions are assumed to be:

$$k_{rl} = \left(\frac{S_c - S_{lr}}{1 - S_{lr}}\right)^3 \tag{I-9}$$

$$k_{rv} = \left(\frac{1 - S_c}{1 - S_{lr}}\right)^3 \tag{I-10}$$

Substituting (I-9) and (I-10) into (I-8) and simplifying yields an explicit expression for the liquid saturation in the steam zone:

$$S_c = \frac{S_{lr} + \psi^{1/3}}{1 + \psi^{1/3}} \tag{I-11}$$

If the steam quality is one, then  $\Psi$  equals 0 and  $S_c$  equals the irreducible liquid saturation,  $S_{lr}$ .

### 3. NAPL Saturation in the Displaced Bank

From multiphase flow theory, equating the pressure gradients in the water and NAPL phases yields an expression for the NAPL saturation in the NAPL bank ( $S_{oc}$ ):

$$-\frac{dP_{w}}{dx} = -\frac{dP_{o}}{dx} \tag{I-12}$$

$$\frac{m_w \mu_w}{\rho_w k_{rw} k} = \frac{m_o \mu_o}{\rho_o k_{ro} k} \tag{I-13}$$

The NAPL mass flux is defined by (I-4). The water mass flux is determined from a mass balance at the steam condensation front and is given by:

$$m_w = m_{inj} + (\rho_l - \rho_v)\phi V_f \left(1 - S_c - S_{oc}\right)$$
 (I-14)

Substituting (I-4) and (I-14) into (I-13) yields:

$$\frac{m_{\rm inj}}{\rho_{\rm w} \phi V_{\rm f}} = \frac{\mu_{\rm o} k_{\rm rw}}{\mu_{\rm w} k_{\rm ro}} \left( 1 + \frac{S_{\rm or}}{S_{\rm oc}} \right) (S_{\rm oc} - S_{\rm or}) - \left( \frac{\rho_{\rm l} - \rho_{\rm v}}{\rho_{\rm w}} \right) (1 - S_{\rm c} - S_{\rm oc})$$
 (I-15)

The right-hand-side of (I-15) is a function of the NAPL bank saturation ( $S_{oc}$ ), steam liquid saturation, and physical constants. The relative permeabilities for this study can be expressed in terms of  $S_{oc}$  with:

$$k_{rw} = \left(\frac{1 - S_{oc} - S_{wr}}{1 - S_{wr} - S_{or}}\right)^3 \tag{I-16}$$

$$k_{ro} = \left(\frac{S_{oc} - S_{or}}{1 - S_{wr} - S_{or}}\right)^{3} \tag{I-17}$$

where  $S_{or}$  is the residual NAPL saturation (i.e., immobile to a waterflood) and  $S_{wr}$  is the irreducible water saturation (i.e., immobile to a NAPL flood). Therefore, it remains only to develop an expression for the condensation front velocity and then (I-15) yields a transcendental expression for  $S_{oc}$ .

#### 4. Steam Condensation Front Velocity

The steam condensation front velocity is determined from an energy balance. The energy injected  $(Q_{inj})$  equals the heat  $(Q_{heat})$  to bring a volume of soil from ambient temperature  $(T_{amb})$  to steam temperature  $(T_{inj})$ . The energy in the injected steam is:

$$Q_{inj} = m_{inj} \left[ X h_v + (1 - X) h_w \right] A_{cs} \Delta t = m_{inj} c_{pw} \Delta T \left[ 1 + \frac{X h_{fg}}{c_{pw} \Delta T} \right] A_{cs} \Delta t$$
 (I-18)

where h is the enthalpy,  $c_{pw}$  is the heat capacity of water,  $h_{fg}$  is the latent heat of vaporization of water,  $\Delta T = T_{inj} - T_{amb}$ ,  $A_{cs}$  is the cross-sectional area to the flow, and  $\Delta t$  is the duration of injection. The energy required to heat a given volume  $(A_{cs} \cdot \Delta x)$  of soil from ambient to steam temperature is:

$$Q_{heat} = \left[ (1 - \phi) \rho_r c_{pr} \Delta T + \phi S_c \rho_w h_w + \phi (1 - S_c) \rho_v h_v \right] A_{cs} \Delta x$$
 (I-19)

$$Q_{heat} = \left[ (1 - \phi) \rho_r c_{pr} + \phi S_c \rho_w c_{pw} + \phi (1 - S_c) \rho_v \left( c_{pw} \Delta T + h_{fg} \right) \right] \Delta T A_{cs} \Delta x \qquad (I-20)$$

Equation (I-20) is derived by noting that in quasi-steady displacement, no NAPL is heated and only the solid, liquid and vapor phases in the steam zone contain heat above ambient conditions. Equating (I-18) with (I-20) and rearranging yields the steam condensation front velocity:

$$V_{f} = \frac{\Delta x}{\Delta t} = \frac{\frac{m_{inj}}{\phi \rho_{w}} \left( 1 + \frac{X h_{fg}}{c_{pw} \Delta T} \right)}{\frac{(1 - \phi)\rho_{r}c_{pr}}{\phi \rho_{w}c_{pw}} + S_{c} + \left( 1 - S_{c} \right) \frac{\rho_{v}}{\rho_{w}} \left( 1 + \frac{h_{fg}}{c_{pw} \Delta T} \right)}$$
 (I-21)

#### B. RESULTS

# 1. MAXIMUM NAPL VISCOSITY FOR A MARGINALLY STABLE STEAM DRIVE

Rearranging equation (I-5) yields an expression for the maximum viscosity of a NAPL which can be driven during steam injection:

$$\frac{\mu_o}{\mu_w} \le \frac{\mu_v \rho_w k_{ro}}{\mu_w \rho_v k_{rv}} \left[ \frac{m_{inj} X}{\rho_w \phi V_f \left( 1 + \frac{S_{or}}{S_{oc}} \right) \left( S_{oc} - S_{or} \right)} \right]$$
 (I-22)

As described above, the relative permeabilities are solely functions of the fluid saturations. Substituting the expression for the condensation front velocity (I-21) into (I-22) yields:

$$\frac{\mu_{o}}{\mu_{w}} \leq \frac{\mu_{v}\rho_{w}k_{ro}}{\mu_{w}\rho_{v}k_{rv}} \left[ \frac{\left(1-\phi\right)\rho_{r}c_{pr}}{\phi\rho_{w}c_{pw}} + S_{c} + \left(1-S_{c}\right)\frac{\rho_{v}}{\rho_{w}}\left(1 + \frac{h_{fg}}{c_{pw}\Delta T}\right)}{\left(\frac{1}{X} + \frac{h_{fg}}{c_{pw}\Delta T}\right)\left(1 + \frac{S_{or}}{S_{oc}}\right)\left(S_{oc} - S_{or}\right)} \right]$$
(I-23)

where  $k_{ro}$  and  $k_{rv}$  are defined by (I-17) and (I-10), respectively. Combining (I-15) with (I-21) and substituting (I-16) and (I-17) yields a transcendental equation the root of which is  $S_{oc}$ :

$$\frac{\mu_{o}}{\mu_{w}} \left(\frac{1 - S_{oc} - S_{wr}}{S_{oc} - S_{or}}\right)^{3} \left(1 + \frac{S_{or}}{S_{oc}}\right) \left(S_{oc} - S_{or}\right) - \left(1 - \frac{\rho_{v}}{\rho_{w}}\right) \left(1 - S_{c} - S_{oc}\right)$$

$$= \frac{\left(1 - \phi\right)\rho_{r}c_{pr}}{\phi\rho_{w}c_{pw}} + S_{c} + \left(1 - S_{c}\right)\frac{\rho_{v}}{\rho_{w}}\left(1 + \frac{h_{fg}}{c_{pw}\Delta T}\right) \qquad (I-24)$$

$$\left(1 + \frac{Xh_{fg}}{c_{pw}\Delta T}\right)$$

Changing the inequality in (I-23) to equal and combining with (I-24) eliminates the NAPL viscosity and yields a single expression for  $S_{oc}$ :

$$X \frac{\mu_{v} \rho_{w}}{\mu_{w} \rho_{v}} \left(\frac{1 - S_{oc} - S_{wr}}{1 - S_{wr} - S_{or}}\right)^{3} \left(\frac{1 - S_{lr}}{1 - S_{c}}\right)^{3} - \frac{\left(1 - S_{c} - S_{oc}\right) \left(1 + \frac{Xh_{fg}}{c_{pw}\Delta T}\right)}{\frac{(1 - \phi)\rho_{r}c_{pr}}{\phi\rho_{w}c_{pw}} + S_{c} + \left(1 - S_{c}\right)\frac{\rho_{v}}{\rho_{w}}\left(1 + \frac{h_{fg}}{c_{pw}\Delta T}\right)} = 1$$
 (I-25)

Recall that the liquid saturation in the steam zone ( $S_c$ ) is determined with (I-11) and  $k_{rw}$  is defined by (I-16). With Sc, (I-25) is then solved for  $S_{oc}$  and the maximum NAPL viscosity for a marginally stable steam drive is calculated with (I-23) or (I-24).

Inspection of equations (I-11), (I-23) and (I-24) reveals the surprising result for the idealized drive that steam injection rate does not influence the maximum NAPL viscosity of the drive. This result occurs because as the steam injection rate increases, the velocity of the NAPL bank increases proportionally. Therefore, the ratio of the vapor to NAPL pressure gradient does not change. This leads to the conclusion that increasing the injection rate does not extend the applicability of a steam drive to more viscous NAPLs. Thus, the primary parameters determining a steam drive of NAPL are the steam injection quality, total soil porosity, irreducible water saturation, and the residual NAPL saturation. Of these parameters, only the steam injection quality lies within the control of the design engineer; the other parameters are fixed by the natural system. For a given system, it may also be possible to alter the viscosity of the NAPL with a pre-

steam surfactant flood but other problems may be created (e.g., foaming when steam vapor contacts surfactant-laden water).

### 2. APPLICATION OF THE MODEL TO OPERABLE UNIT ONE, HILL AFB

The physical and thermal properties of water and the soil matrix found at OU-1 are listed in Table I-1. Recall, the density of water was assumed to be the same at injection and ambient temperatures in the model development.

TABLE I-1 PROPERTIES FOR MODELING					
Water and Steam Properties					
Liquid Density (kg/m^3)	995				
Vapor Density (kg/m^3)	0.6				
Water Viscosity at T <sub>amb</sub> (cp)	1.0				
Water Viscosity at Tini (cp)	0.282				
Vapor Viscosity at T <sub>inj</sub> (cp)	0.0126				
Water Heat Capacity (J/kg/K)	4,190				
Heat of Vaporization (J/kg)	2,257,000				
Injection Temperature (°C)	100				
Matrix Properties					
Solid Density (kg/m^3)	2,650				
Solid Heat Capacity (J/kg/K)	1,000				
Total Porosity (%)	38				
Irreducible Water Saturation (%) 10					
Residual NAPL Saturation (%) 5					
Ambient Temperature (°C) 15					

Using the properties listed in Table I-1, the maximum NAPL viscosity allowing displacement by steam injection is plotted in Figure I-3 as a function of the steam injection quality. The figure illustrates an optimum injection quality of about 0.43 which yields a maximum NAPL viscosity of about 2.5 centipoise (cp). The NAPL at Operable Unit One has a viscosity significantly higher than 2.5 cp and therefore is not expected to be displaced. In addition, the steam injection during the field study was maintained at a quality close to one which has a maximum NAPL viscosity of about one cp for stable displacement.

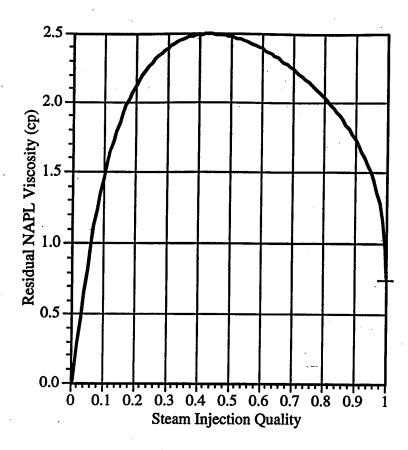


Figure I-3. Maximum NAPL Viscosity for Stable Steam Displacement

#### C. CONCLUSIONS

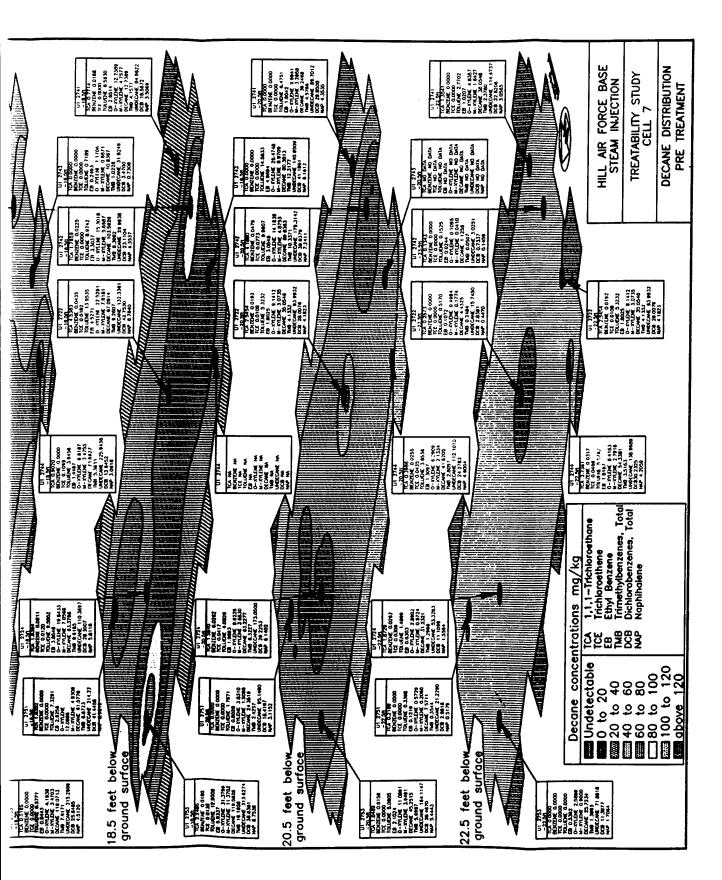
The model presented in this appendix indicates that residual NAPL found at Operable Unit 1, Hill Air Force Base, Utah will not be displaced by steam injection because of its relatively high viscosity. This result was observed in the field demonstration. The primary recovery mechanism is then expected to be distillation of the NAPL components which was also observed. General results from the model indicate a residual NAPL with a viscosity greater than 2 or 3 centipoise will not be driven by steam injection. This implies hydrocarbon mixtures such as diesel (~11 cp) and fuel oil No. 2 (~7 cp) will not be displaced while lighter mixtures such as gasoline (~0.4 cp) will be mobilized. Kerosene (~3 cp) is an intermediate hydrocarbon mixture with questionable mobilization potential. Chlorinated hydrocarbons such as trichloroethylene (~0.6 cp) which makeup dense non-aqueous phase liquids (DNAPLs) generally have favorable viscosities for a steam drive.

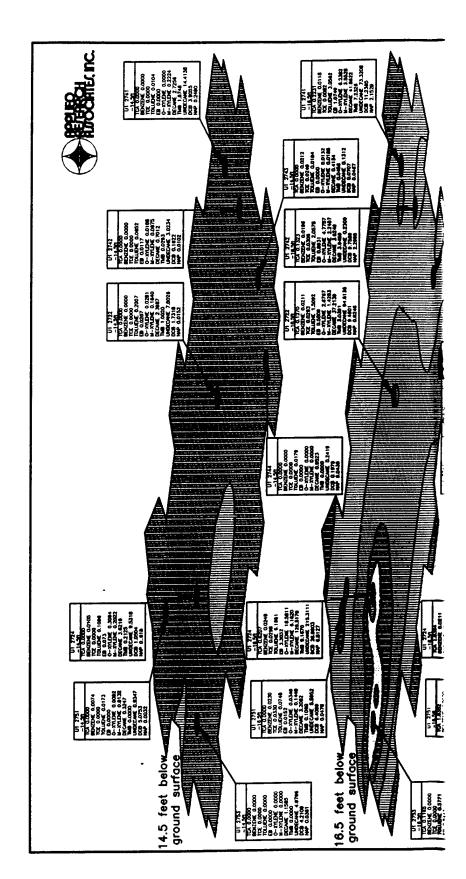
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### APPENDIX J

CONCENTRATIONS OF TARGET ANALYTES DETECTED

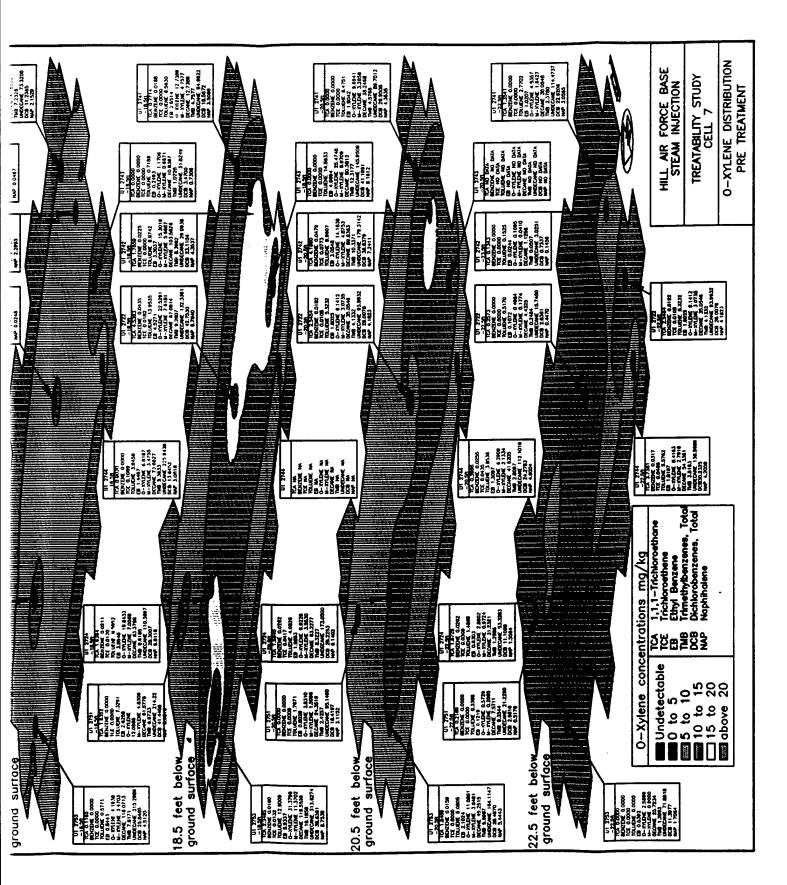


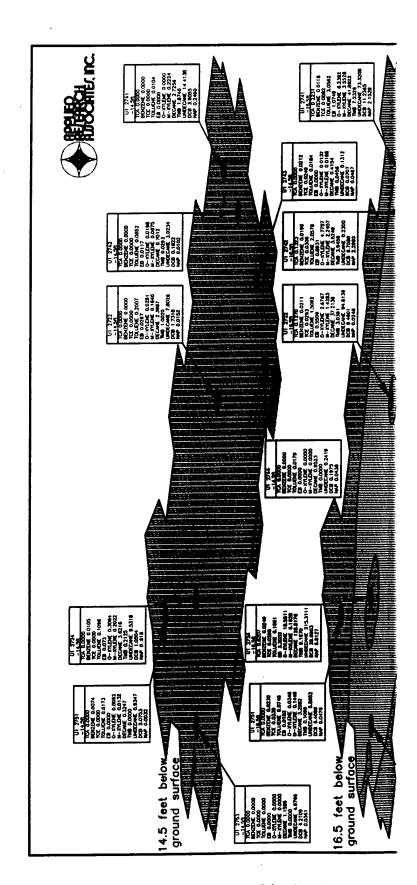


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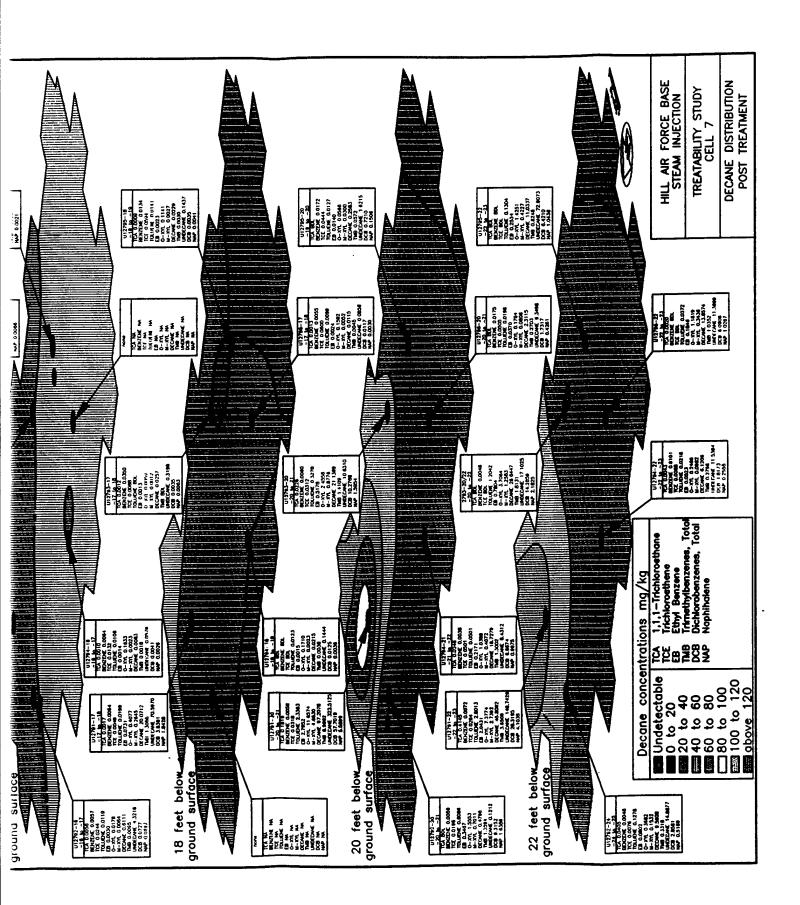
Figure J-1. Pretreatment Distribution of Decar

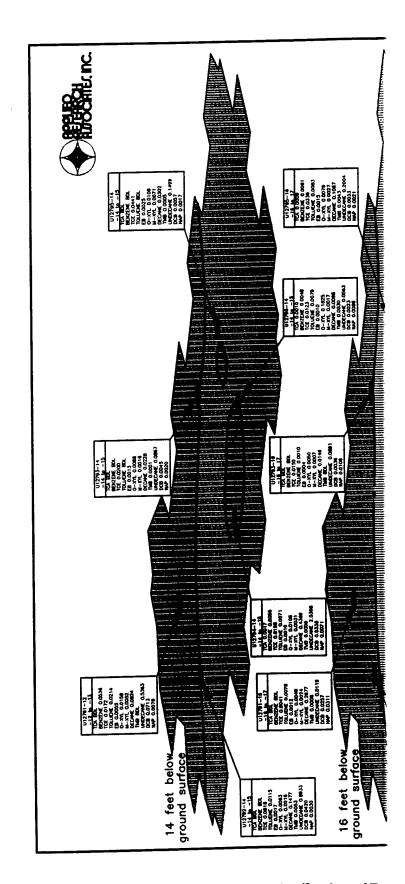




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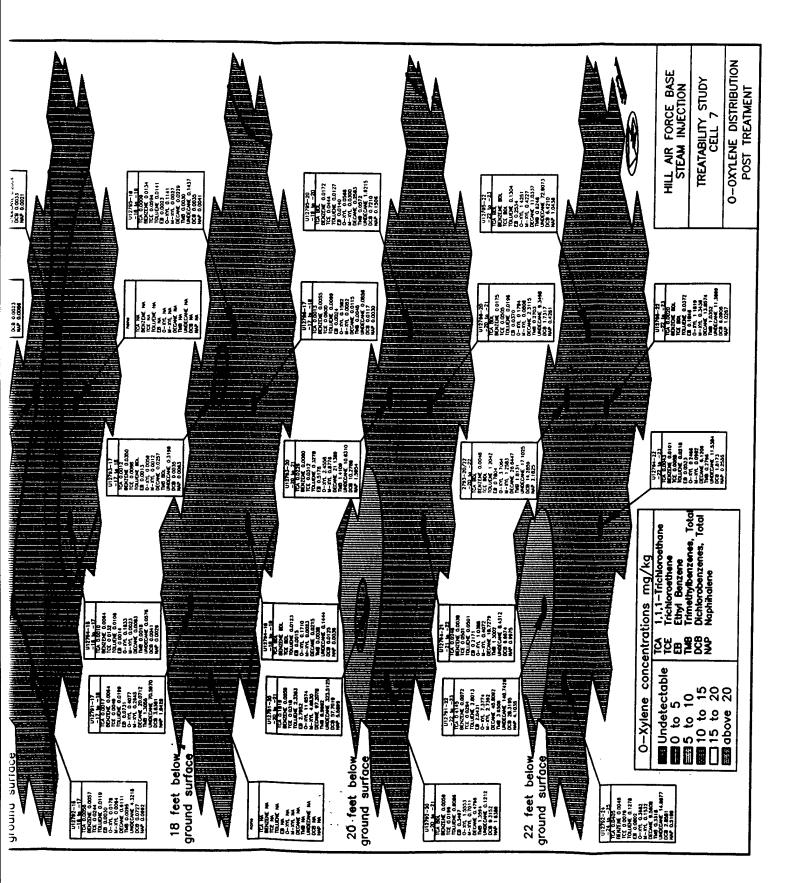
Figure J-2. Pretreatment Distribution of o-Xyl





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Figure J-3. Posttreatment Distribution of De



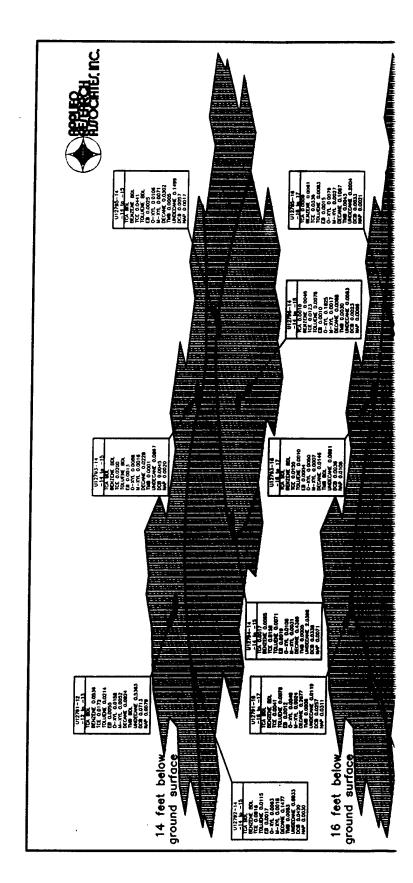


Figure J-4. Posttreatment Distribution of o-Xy.